METALLURGIA

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Vol. 55 No. 327

JANUARY, 1957 JAN

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METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER"

JANUARY, 1957

Vol. LV. No. 327

Oxygen in Steelmaking

If one were asked to say which element plays the most important role in the metallurgy of iron and steel, it would not be easy to arrive at a clear-cut decision. Carbon, with its enormous influence on the structure and mechanical properties of both iron and steel has strong claims to the title, whilst hydrogen, with its harmful effects, cannot lightly be dismissed. Again, nitrogen, phosphorus, sulphur, silicon and manganese-with their influence for good or ill—must all be considered. There is one element, however, which is closely connected with ferrous metals from the cradle to the grave-so to speak-namely, oxygen. In most ore deposits, iron is associated with oxygen as the oxide or carbonate-which becomes oxide on calcining-and the operations in the blast furnace are concerned with the separation of the two. At the next refining stage, oxygen is associated with the removal of impurities in steelmaking, and its presence in the liquid metal can be used in the prevention of piping in steels for certain purposes. In the solid steel, oxygen is mainly present in the form of nonmetallic inclusions, whose form and distribution may influence considerably the mechanical properties. Finally, the enormous wastage which takes place each year as a result of rusting is a further instance of the important part oxygen plays in the metallurgy of iron

The ores of iron were formed in prehistoric times, and the association of oxygen with iron in rusting must go back to the beginnings of ferrous metallurgy. Even in process metallurgy oxygen, in the form of iron oxide, or associated with nitrogen in air, has long been used in refining, but it is only during the last one or two decades that oxygen itself has been used in iron and steelmaking operations. As Mr. D. J. O. Brandt pointed out recently in a paper to the West of Scotland Iron and Steel Institute, it was possible in the years immediately following the end of World War II to cover the use of oxygen in iron and steel production in a reasonable measure of detail in the course of a single paper. Some measure of the change which has taken place in the last ten years is given by the fact that the first book* dealing exclusively with the technology of oxygen in the iron and steel industry was published a few months ago. Developments in this field are taking place all over the world, particularly on the Continent, and although economic considerations have been unfavourable in the United Kingdom, the position is changing and the near future should see an appreciable increase in the use of oxygen in this

To date, the most successful uses of oxygen have been in the steelmaking field. The case for the use of oxygen-enriched blast in iron making is not yet established, for although it should result in faster and more thermally efficient blast-furnace operation, its use inevitably

results in a higher tuyere zone temperature, with attendant operating difficulties. The nitrogen performs a useful function in transferring heat from the tuyere zone to the solid material in the upper part of the stack. There appears, however, to be some advantage in using oxygenenriched blast in the low-shaft furnace, which, although still suffering from teething troubles, may have an important part to play in making possible the use in ironmaking of fuels other than the high strength coke demanded by the orthodox blast furnace.

With the general deterioration in ore quality and shortage of steel scrap, an ever increasing burden is being placed on steelmaking plants. Because of this, coupled with the continued demand for higher steel output, methods of maintaining or increasing production without heavy capital expenditure are constantly being sought. Oxygen has proved extremely useful in this connection, and has been successfully used on a production basis in several different applications. The desiliconisation of blast-furnace metal by injecting oxygen into it in the ladle is well-established on the Continent. and is finding increasing application in this country. A recent development, in which a measure of dephosphorisation is achieved by extending the period of oxygen injection and making additions of lime or limestone, may reduce still further the metallurgical load on the steelmaking plant.

In steelmaking by the converter process, there are a number of ways in which oxygen may be used to advantage. In addition to increased operating rates, enrichment of the blast to 30% or 35% oxygen enables steels with nitrogen contents as low as 0.004 to 0.005% to be made, whilst with a blast of a mixture of oxygen and carbon dioxide or superheated steam deep-drawing quality steels containing less than 0.0025% nitrogen are being produced. It was this need to produce low nitrogen steels for deep drawing which gave rise to much of the work on oxygen enrichment.

Again with a view to producing high quality Bessemer steels, a process has been developed in Austria, known as the L–D (Linz-Donawitz) process, which employs a blast of pure oxygen directed on to the surface of the metal from above. The reactions are conducted through a fluid slag formed on the surface, and production rates are extremely high. The process is not satisfactory for treating irons containing much more than 0.2% phosphorus, but a modification to enable it to convert 2% phosphorus iron is at present under development.

The advantages to be gained by the use of oxygen in the final stages of decarburisation in the open hearth furnace include savings in time, fuel and feed ore, and improved slag control and desulphurisation. Difficulties with violent agitation and excessive fume are encountered if attempts are made to use oxygen whilst the bath analyses more than $0\cdot4\,\%$ carbon. The use of oxygen to improve the combustion of the fuel, which was at one

(Continued on page 10)

Oxygen in Iron and Steelmaking "by J. A. Charles, W. J. B. Chater and J. L. Hanson. 1956, Butterworth Scientific Publications, London.

Meeting Diary

2nd January

Institute of Welding, Manchester and District Branch. "Hydrogen—Barrier to Welding Progress," by C. L. M. COTTRELL. College of Technology, Manchester. 7.15 p.m.

3rd January

Incorporated Plant Engineers, Peterborough Branch. "Dust Control," by R. J. Prer. White Lion Hotel, Church Street, Peterborough. 7.30 p.m.

Institute of Metals, Birmingham Local Section. Discussion on "Timber versus Metals." Opening Speakers: D. O. REECE, W. E. BALLARD, DOROTHY PILE. Birmingham Exchange and Engineering Centre, Stephenson Place, Birmingham. 6.30 p.m.

Institute of Metals, London Local Section. "The Metallurgical Challenge of Nuclear Power," by Dr. H. K. Hardy. 17, Belgrave Square, London, S.W.1. 6.30 p.m.

Leeds Metallurgical Society. "Some High-Temperature Properties of Copper Alloys," by Dr. J. P. Dennison. Large Chemistry Lecture Theatre, The University, Leeds, 2. 7.15 p.m.

4th January

Institution of Mechanical Engineers. "An Experimental Investigation of the Process of Expanding Boiler Tubes," by Dr. J. M. ALEXANDER and PROF. H. FORD. 1, Birdcage Walk, Westminster, London, S.W.1. 6 p.m.

8th January

Incorporated Plant Engineers, Edinburgh Branch. "Electronics in Industry," by R. B. SMITH. 25, Charlotte Square, Edinburgh. 7 p.m.

Institute of Metals, Oxford Local Section. "Gases in Metals," by Dr. C. E. Ransley. Cadena Cafe, Cornmarket Street, Oxford. 7 p.m.

9th January

Incorporated Plant Engineers, Western Branch. "Factory Electrical Distribution Systems," by R. H. Cobbold. Grand Hotel, Bristol. 7.15 p.m.

Institute of Fuel, North-Western Section. "Liquid Fuels for Industrial and Domestic Boilers," by G. J. Parker. Engineers Club, Albert Square, Manchester. 2.30 p.m.

Institute of Marine Engineers, Scottish Section. "Cast Irons in Marine Engineering," by E. M. CURRE, Small Hall of Institution of Engineers and Shipbuilders, 39, Elmbank Crescent, Glasgow, C.2. 7.30 p.m.

Institution of Production Engineers, Liverpool Section. "Cutting Tools—Application and Research," by M. O. Short. Bradford Hotel, Tithebarn Street, Liverpool. 7.30 p.m.

Society of Chemical Industry, Corrosion Group. "Corrosion Problems in Nuclear Power Production," by A. B. McIntosh. Society of Chemical Industry, 14, Belgrave Square, London, S.W.I. 6.30 p.m.

10th January

East Midlands Metallurgical Society. "The Use of Radioactive Isotopes in Industry," by Dr. A. D. Merriman. Nottingham and District Technical College. 7.30 p.m.

Institute of British Foundrymen, Beds./Herts. Section. "Reduction of Dust in the Production of Loose Pattern Moulds by Core Assembly," by E. H. BEECH. W. H. Allen, Sons & Co., Ltd., Bedford. 7.30 p.m.

Institution of Mechanical Engineers. Discussion on "Diagnosis of Causes of Plain Bearing Failure." 1, Birdcage Walk, Westminster, London, S.W.1. 6.45 p.m.

Liverpool Metallurgical Society. Student Prize Competition. Joint Meeting with the University of Liverpool Metallurgical Society. 'Zhe University, Liverpool. 7 p.m.

11th January

Institute of Physics, Manchester and District Branch. "Ferromagnetism and Magnetic Materials," by A. E. DE BARR. Bragg Building, Manchester University. 6.45 p.m.

15th January

Institute of British Foundrymen, East Anglian Section.
"Foundry Developments," by D. Killingworth. Lecture
Hall, Public Library, Ipswich. 7.30 p.m.

Institute of British Foundrymen, Slough Section. "Coreblowing," by G. W. Fearfield. Lecture Theatre, High Duty Alloys, Ltd., Slough. 7.30 p.m.

Institute of Metals, South Wales Local Section. "Fatigue," by Major P. Litherland Teed. Dept. of Metallurgy, University College, Singleton Park, Swansea. 6.45 p.m.

16th January

Institute of British Foundrymen, Southampton Section. Technical Film Show—"The Kitimat Story" (production of aluminium) and "Aluminium Casting." Technical College, St. Mary's Street, Southampton. 7.30 p.m.

Manchester Metallurgical Society. "Physical Chemistry in Extraction Metallurgy," by Dr. F. D. RICHARDSON. Manchester Room, Central Library, Manchester. 6.30 p.m.

17th January

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Incorporated Plant Engineers, Blackburn Branch. "Modern Chainmaking Techniques," by W. Gibson Biggart. Golden Lion Hotel, Blackburn. 7.30 p.m.

Institute of Fuel, North-Western Section. "Refractories for the Industrial Boiler and Furnace," Morgan Refractories, Ltd. Liverpool Engineering Society, 9, The Temple, Dale Street, Liverpool. 7 p.m.

Institute of Metals, Birmingham Local Section. "Some Metallurgical Aspects of a Gas Turbine Engine," by S. HESLOP. Birmingham Exchange and Engineering Centre, Stephenson Place, Birmingham. 6.30 p.m.

18th January

Incorporated Plant Engineers, Birmingham Branch. "Are Welding": lecture and film presented by F. A. Thomas. Imperial Hotel, Temple Street, Birmingham. 7.30 p.m.

21st January

Institute of Metals, Sheffield Local Section. "Research Work in the Department of Metallurgy," by Prof. A. G. QUARBELL. Engineering Lecture Theatre, The University, St. George's Square, Sheffield, 1. 7.30 p.m.

Institution of Production Engineers, Coventry Section. "Precision Casting," by Dr. D. F. McVITTE. (Paper will include reference to Shell Moulding and Investment Casting). The Craven Arms, High Street, Coventry. 7. p.m.

North East Metallurgical Society. "The Industrial Development of Tees-Side," by J. C. H. BOOTH. Constantine Technical College, Middlesbrough. 7.15 p.m.

22nd January

Institution of Production Engineers, Tees-Side Section.
"Precision Casting," by J. S. Turnbull. Cleveland Scientific and Technical Institution, Corporation Road, Middlesbrough. 7 p.m.

24th January

Society of Chemical Industry, Corrosion Group. Conversazione. Members are invited to submit exhibits illustrating their researches. Battersea Polytechnic, Battersea Park Road, London, S.W.11. 6.30 p.m.

30th January

Institute of British Foundrymen, London Branch. "High-Duty Iron Castings Production," by C. R. VAN DER BEN. Constitutional Club, Northumberland Avenue (south-east from Trafalgar Square), London, W.C.2. 7.30 p.m.

Manchester Metallurgical Society. "The Preparation of Germanium and Silicon for Use in Semiconductor Devices," by S. E. Bradshaw. Manchester Room, Central Library, Manchester. 6.30 p.m.

31st January

Incorporated Plant Engineers, Sheffield and District Branch. "The Care and Maintenance of Wire Ropes": British Ropes Ltd., Grand Hotel, Sheffield. 7.30 p.m.

Institute of Metals, Birmingham Local Section. "Pure Metals," by Dr. J. C. Chaston. Birmingham Exchange and Engineering Centre, Stephenson Place, Birmingham. 6.30 p.m.

The Application of Induction Heating to the Surface Hardening of Steel

By J. Hamilton, B.Sc. (Eng.), Grad. I.E.E.

Research Laboratory, Birlec, Ltd.

The use of high frequency induction heating in the surface hardening of steel has been gaining favour in recent years. Following an outline of the principles underlying induction heating, the author discusses aspects affecting its practical application and the types of generator available. He concludes with a description of a few typical installations.

THE need for a hard surface supported by a relatively soft core commonly arises in the design of steel components for engineering applications. There are two main approaches to this problem. The first is to make additions to the surface layer (by carburising, nitriding, etc.) which is thereby preferentially hardened; the second is to use a hardenable steel and locally heat treat the surface layer. To do this it is necessary to heat the surface so rapidly that only a thin layer reaches hardening temperature and the core is left relatively cool, and thus remains soft after quenching. This rapid heating requires a high rate of energy input to the steel, and a convenient means of achieving this is to induce an electric current to flow in a shallow surface layer which is heated by virtue of its resistance to the flow of current.

Principles of Induction Heating

Induction heating may be considered to be a special case of the transformer, the construction and operation of which are well known. If a current is passed round a helical coil it sets up a magnetic field within the coil. When this current is alternating the resulting magnetic field will induce circulating currents in any electrical conductor which is placed within the coil and these currents will flow in the opposite direction to the coil current. If one considers a cylindrical workpiece to be made up of a series of thin concentric tubes, the current induced in each will depend on the magnetic field within this tube. This is weaker than the external magnetic field by virtue of the current flowing in opposition to the coil current, and thus each tube is subjected to a weaker magnetic field than the surrounding tubes, i.e., in a solid cylinder, the field strength and, consequently, the current density, will be highest near the surface and will fall away as the centre is approached.

High frequencies induce relatively higher currents, so that the weakening effect becomes more marked at higher frequencies, and the current density falls more rapidly. The fall of current density obeys a logarithmic law, but the effective current may be taken as one of uniform density flowing in a layer of depth

$$p = \frac{1}{2\pi} \sqrt{\frac{\rho}{\mu f}}$$
 cm.

where $\rho=$ resistivity in e.m.u., $\mu=$ magnetic permeability, and f= frequency of the alternating current. This property of high frequency currents is known as "skin effect" and the value p is known as the "penetration depth."

The phenomenon of skin effect, which constrains alternating currents to flow near to the surface of a

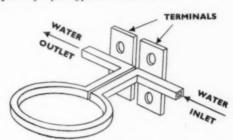


Fig. 1.—Elementary single turn inductor for heating cylindrical work piece.

conductor, is the essential factor in the application of induction heating to the surface hardening of steels. If a sufficiently high current is induced in the component being treated, the heat generated in the surface layer can heat this layer to hardening temperature before any appreciable heat flow to the inner core takes place. To achieve the desired heating rate, a power input of up to 25 kW./sq. in. may be required, and for such high power densities it is desirable to use a single-turn coil, as shown in Fig. 1.

There are several points in favour of a single-turn as distinct from a multi-turn coil. For any particular duty, the voltage applied to a coil is proportional to the number of turns, and the voltage should be kept low, both in the interests of safety to the operators and simplicity of construction. For high efficiency and accurate control of the extent of the heated zone, the space between work and coil should be kept to a minimum consistent with prevention of arcing across this space. A convenient clearance for single turn inductors is between in and Coils must be water cooled and multi-turn coils of small gauge tube would require very high water pressures, and would easily be blocked by deposits from the water. A single-turn inductor can be machined from a comparatively massive block of copper, so giving a robust construction which is much better able to withstand careless handling than a coil of tubing. All these factors indicate that a single turn operating at a relatively low voltage is a desirable type of inductor.

It is generally easier to generate and distribute high frequency power at higher voltages and lower currents than are required by single-turn coils, so a step down transformer with a ratio of between about 10:1 and 20:1 is required to supply the inductor. The single turn coil or inductor surrounds the work and carries an alternating current which induces a corresponding current in the work. Heat generated in the inductor is

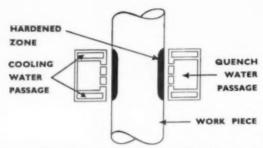


Fig. 2.—Diagrammatic section through inductor with provision for spray quenching.

wasted, and it is therefore desirable to construct inductors of high conductivity copper to minimise the power lost in this way. Even in a copper inductor there will be some power loss, so passages are provided in the inductor through which cooling water is passed. In many cases water quenching of the work may be employed, and additional ducts must be provided to supply quench water to a series of jets in the inner wall of the inductor. It is common practice to apply the quench water automatically at the end of the heating period, although for small components it is sometimes more convenient to dispense with spray quenching and drop the work into a tank of water after heating.

Practical Applications

It is frequently necessary to surface harden components (e.g., long shafts) where the area is so large that an inconveniently high power would be required. A simple means of overcoming this difficulty is to heat only a narrow band round the shaft, which is moved at a constant speed through the inductor and emerges with the surface heated to the desired temperature. As it leaves the inductor the heated shaft is quenched by being passed through a water spray. The heating and quenching is a continuous process, so that there is no theoretical limit to the length of component which may be treated in this way. Where long shafts are being progressively hardened by this method, it is necessary to minimise distortion if a straightening operation is to be avoided. This can be achieved by rotating the shaft during its travel through the heating and quenching zones.

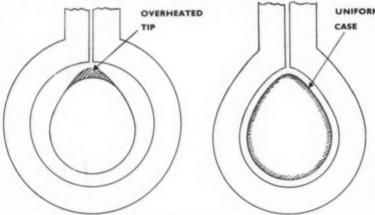


Fig. 3.—Effect of inductor design on case uniformity when hardening a cam profile.

this means a straight hardened product is obtained, and only a small grinding allowance is necessary. In addition to this advantage, rotation enables a very uniform case depth to be obtained. So far only cylindrical work has been mentioned, as this provides a simple illustration of the induction hardening process, but it is in no way limited to this shape. Suitable inductors can be designed to harden flat or even spherical surfaces, and the profiles of irregular objects such as cams or gear teeth can also be treated.

Figs. 1 and 2 show very elementary arrangements for hardening a cylindrical workpiece, but more complex processes can now be considered. When an irregular workpiece, e.g., a valve cam, is heated in a circular inductor, those parts of the work nearest to the inductor tend to be overheated. In mild cases this results in an increased case depth on projections and corners, and in severe cases cracking or incipient melting may occur. Careful inductor design can overcome this difficulty by arranging for a uniform clearance between work and inductor as shown in Fig. 3.

The beneficial results of hardening gear teeth are well appreciated, and induction heating provides a most convenient method of hardening teeth to just below the roots, leaving the body of the gear relatively soft. For this purpose it is seldom practicable to use an inductor shaped to the profile of the gear teeth, as it is usually necessary to rotate the gear during heating, to produce a uniform depth of hardening and to prevent distortion -a most important point where gears are concerned. Uniform heating of the teeth can be obtained by careful choice of frequency. Very high frequency current will overheat the tips, as already explained in connection with cam hardening, while a very low frequency, where the current penetration depth is large compared with the tooth size, will prevent the current from following the tooth contour, so that most of the heat is generated near the roots, with consequent overheating at that point. An intermediate frequency can be found which will uniformly heat the body of the tooth, giving a hardened pattern as shown in Fig. 4c.

Tubular components, such as gudgeon pins and rocker shafts, where the outer surface only is to be hardened, are suitable subjects for the induction hardening process. For long tubes such as rocker shafts, a certain amount of flexibility is desirable, and it is a simple matter to harden

only the bearing surfaces, power being UNIFORM switched off while the inductor passes over the intermediate zones, which are thus left soft. This method enables any desired pattern of hard and soft zones to be easily obtained, and eliminates the need for copper plating or any similar precautions which would be required in conjunction with a carburising process. In surface hardening the outside of tubes, it is not normally possible to obtain a case depth greater than about one third of the wall thickness. Any attempt to obtain a greater depth results in through hardening, as a sufficient temperature gradient cannot be maintained without recourse to special precautions, such as water cooling of the bore.

In using induction heating for surface hardening, it is not always

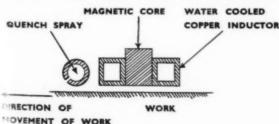
possible to use the simple, and very efficient, enveloping inductor which has been considered so far. For parts which have profiles other than circles or approximate circles, it is necessary to use inductors of more complex design. Take, for example, the hardening of a flat surface. A single turn inductor placed close to the surface will heat a circular band under the inductor leaving a cold spot in the centre. Nearly uniform heating can be obtained by using a flat spiral, "pancake" coil. Where a large flat area is to be surfaced hardened the progressive method may be employed using a "hairpin" This consists of two parallel conductors arranged in the form of a "hairpin," which scans the surface of the work followed by a spray quench. Inductors of this type are frequently less efficient than the enveloping type. To maintain the maximum possible efficiency, a core of ferromagnetic material may be introduced into the magnetic circuit, as in Fig. 5. This core performs a function similar to the laminated iron core of a transformer, in that it reduces the magnetising current required, and, consequently, reduces the copper losses in the inductor.

Several alternative materials are available for use as cores. The simplest type consists of laminated silicon steel of similar construction to a conventional transformer core. This type is only suitable at frequencies up to about 10,000 c./sec., as higher frequencies would require inconveniently thin laminations. Laminated material must be used to restrict the flow of induced current in the core, and so avoid overheating. Alternative materials which are particularly suited for use at radio frequencies are finely divided iron or nickel-iron alloy powder, insulated with a synthetic resin and pressed into a solid block, and "ferrites," which are compounds of the ferric oxide of iron with oxides of other metals, notably copper, manganese, magnesium, nickel, zinc and ferrous iron. As the ferrites are insulators (with resistivities of the order of 105 ohm-cm.) they can safely be used in high frequency magnetic fields without risk of heating due to eddy currents.

In some cases it may be impossible to insert the work into a simple inductor. This is the case, for example, when the bearing surfaces of a crankshaft are to be hardened. For such operations it is possible to use an inductor split into two sections which, when clamped together form a complete circuit, as shown in Fig. 6. It is usual to make the contact faces of some material which does not easily oxidise, such as silver. It is, of course, important to open and close the inductor with the high frequency power switched off.

Power and Heating Time

The "penetration depth" (which can be taken as the



5.—Diagrammatic section through hair pin inductor surfaces.

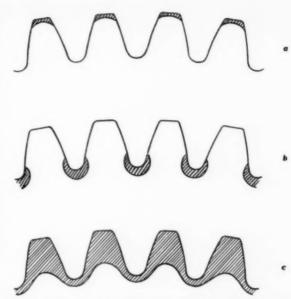
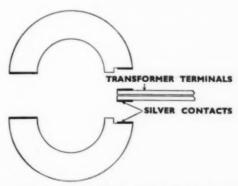


Fig. 4.—Effect of frequency on gear hardening: frequency too high; (b) frequency too low; (c) frequency correct.

depth of the surface layer in which about 90% of the power is generated) depends mainly on the permeability and resistivity of the material being heated and on the frequency of the current. Most hardenable steels lose their ferromagnetic properties at about 720°C., so that there is a sudden change in penetration depth when this temperature is reached. For most steels the penetration depth is $0.08/\sqrt{f}$ in. at 20° C., and $20/\sqrt{f}$ in. at 800° C., where f is the frequency of the applied current. In raising the steel to hardening temperature this change takes place during the heating period, and calculation of the effective penetration depth becomes a complex mathematical process. However, for practical purposes, it is sufficiently accurate to consider that the heat is generated in a layer of depth $2/\sqrt{f}$ in. The heat will penetrate beyond this to a depth depending on the heating time, adjustment of which provides a convenient means of controlling the hardened case depth. Precise mathematical analysis of the heat flow is rather complex, but for induction



magnetic core for progressive hardening of flat Fig. 6.—Diagrammatic view of split inductor in the open position.

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Fig. 7.—Front view of 15 kW. self-contained induction heater.

heating purposes it is a useful guide to take $0.04\sqrt{t}$ in. as the depth to which heat will flow beyond the layer in which it is generated, where t is the heating time in seconds. We now have a simple means of estimating the hardened depth in terms of the frequency and heating time, by adding together the depth of the layer in which heat is generated and the depth to which heat conduction is effective. Thus the hardened depth is given by

$$D = 2/\sqrt{f} + 0.04\sqrt{t}$$
 in.

where f is the frequency in cycles per second and t is the heating time in seconds.

The power required for surface hardening depends mainly on the hardened depth and the area to be treated in a given time. Accurate methods of calculating the power requirement for a particular job are quite b yond the scope of this article, as there are many factors which influence the electrical efficiency of the inductor and the heat lost by conduction in the workpiece. The following equation is a helpful guide to the energy required

$$E = 320 DA \text{ kW. sec.}$$

where E= energy required, D= hardened depth, and A= hardened area.

The conditions of time and power obtained by these simplified expressions are only approximate, as such general formulae cannot take into consideration all the conditions arising in any particular case. The power values so obtained are only average figures, and more power is necessary where conditions require a type of inductor which is electrically inefficient, or where thermal losses are unusually high. Similar results may sometimes be obtained by using increased power and decreased time, or vice versa, so that the expressions for case depth and power are only an indication of typical conditions, and do not represent the only possible condition.

Various practical considerations impose limits on the range of case depths available. For example, the current

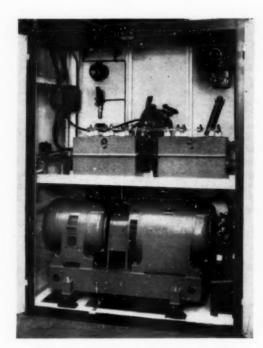


Fig. 8.-Rear view of the same machine.

penetration fixes a limit to the minimum case depth obtainable, and for 3,000, 10,000 and 400,000 c./sec., the minimum case depths which can normally be obtained are about, 0.06 in., 0.04 in. and 0.015 in., respectively. Even if sufficient power is available to heat the surface almost instantaneously, a finite time (of the order of 0.1 sec.) is necessary in practical applications for solution of the constituents of the steel. Thus it is impossible, even with very high frequencies, to obtain a case depth less than about 0.010 in. By using a frequency of 500,000 c./sec. this theoretical minimum is increased by only about 30%, so there is little to be gained by using any higher frequency for surface hardening.

Generating Equipment

The available sources of high frequency power fall into three main classes: rotary motor driven alternators; valve oscillators; and spark gap oscillators. The latter cannot normally be controlled with sufficient accuracy or stability for surface hardening duty, but both motoralternators and valve oscillators are well suited to this process.

High frequency alternators are used for generating frequencies up to about 10,000 c./sec., and are available in sizes ranging from a few kilowatts up to several hundred kilowatts. The design of such machines becomes increasingly difficult as operating frequency rises, and the upper economic limit is about 10,000 c./sec., in the interests of conversion efficiency and simplicity of design. Alternators may be divided into two main classes: homopolar and heteropolar. It is not necessary to go into details of the design here, but there is one difference in characteristics between the types which is important in relation to induction heating. In the heteropolar machine the output responds rapidly to changes in excitation voltage, while with the homopolar construction there is an appreciable time delay, which

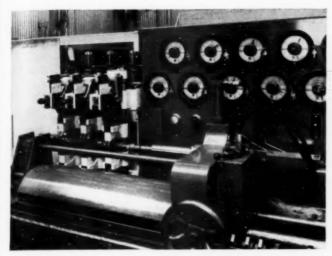


Fig. 9.-Machine for surface hardening automobile camshafts.

may be several seconds from zero to full voltage. This difference in characteristics makes the heteropolar type more attractive for induction hardening applications, where heating time must be accurately controlled and frequent and precise switching is necessary, as it is much more convenient to employ field switching than direct switching of full alternator output.

Valve oscillators for surface hardening commonly operate at about 400,000 c./sec., and are available with outputs up to about 160 kW. These generators are usually triode oscillators of the Hartley or Colpitts type. As has already been mentioned, a frequency of 400,000 c./sec. enables case depths only slightly more than the minimum possible to be obtained, so there is little to be gained by working at a higher frequency, which only complicates the problems of generation and utilisation.

The first consideration in deciding which type of generating equipment should be used is the technical aspect of the problem. It has already been shown that for any frequency there is a minimum case depth which can be obtained, and if shallow cases, for example 0.025 in., are required it is necessary to use a valve oscillator, while case depths of about 0.060 in. and upwards can conveniently be obtained at 10,000 c./sec. on parts of fairly large size, say one inch diameter.

For "single shot" surface hardening, the heating time decreases and power input increases as the frequency is reduced, so that in some cases a valve generator can do the work of an alternator of much larger capacity. This can best be illustrated by the example of 1 in. diameter journal being hardened to a depth of 0.060 in. for a length of 1 in.

From $D = 2/\sqrt{f} + 0.04\sqrt{t}$ we obtain

t=2 sec. for 400,000 c./sec. valve oscillator, or 1 sec. for a 10,000 c./sec. alternator.

The energy input = 320 DA kW. sec.

= $320 \times \pi \times 0.75 \times 0.060$ kW. sec.

= 45 kW. sec.

Thus the power requirement is 45 kW. for 1 sec. from the alternator, or 221 kW. for two sec. for the valve oscillator. Provided 2 sec. heating time is acceptable



Fig. 10.—Multi-purpose induction hardening machine with rotating turret to carry four work-holding jigs.

from a production point of view, the valve oscillator would be the better choice for this particular job.

Typical Installations

Figs. 7 and 8 show the front and rear views of a selfcontained induction heater comprising a 15 kW., 8.5 kc./ sec. motor alternator set; power factor correction capacitors; alternator control gear; and an output transformer giving 200 kVA at voltages down to about 15 V to suit a single turn inductor. It is interesting to note that this represents a power factor of 0.125, which is not uncommon for induction heating coils, hence the need for power factor correction capacitors in high frequency alternator circuits.

The machine shown in Fig. 9 has been designed for surface hardening automobile camshafts, and is supplied with power by a 50 kW. valve generator. In the illustration the covers for the three split inductors and their closing mechanism have been removed: the inductor on the right is shown closed. The work supporting carriage is indexed by hand against automatic stops which locate the cams and bearings in turn in the appropriate inductor, the heating and quenching sequence being controlled by the bank of timers on the right hand panel. Automatic rotation of the shaft ensures that each cam is correctly aligned with the inductor.

Four different work-holding jigs may be mounted on the square turret seen in the illustration of the multipurpose hardening machine (Fig. 10), and any one brought into line with the workhead unit, which has provision for quick interchange of inductors. The workhead unit may be raised and lowered at controlled speed for progressive hardening, or automatically stopped in

any position for single shot hardening.

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George James Snelus

An Early Student of Metallurgical Science

By Eric N. Simons

Much early work on metallurgical processes was of an empirical nature. In many cases discoveries were made and put into practice with little understanding of the precise mechanism of operation. George Snelus entered the steel industry with a scientific training, and although his name may be less familiar than those of Huntsman, Bessemer, Thomas and Siemens, he played an important part in establishing the scientific principles behind the basic process of steelmaking.

IT is rather remarkable that many of the spectacular developments in iron and steel manufacture are due to men outside the industry. Hunstman, Bessemer,

Thomas and Siemens were not steel men, but the processes known by their names have played important roles in the industry's progress. On the whole, the industry has not been slow to take up these inventions, and, having done so, to learn as much as possible about the factors governing them. The subject of this article, George James Snelus, was a professional metallurgist and for him is claimed the distinction of being the first to discover the principle of the basic process of making steel-a discovery based on observation and experiment.

Scientific Training

Snelus was born in Camden Town, London, on June 24th, 1837, the son of James Snelus, a builder, who died before the boy was twelve years old. He received a good education, however, being given the training considered in those days adequate for one intended

to be a teacher. Nevertheless, teaching was not a profession that appealed to the boy, whose main interests lay in the direction of applied science. Accidentally, he became interested also in chemistry and metallurgy.

Educated at St. John's College, Battersea, Snelus one evening turned up at the Polytechnic to hear a lecture on the recently-invented Bessemer process, to be delivered by Professor Pepper. The clear and fascinating exposition of this revolutionary discovery fired Snelus's imagination, and he determined to apply himself to science so that he might ultimately make a position for himself in the steel industry.

After a little preliminary instruction he became a student under Professor Roscoe at Owens College, Manchester. Later, he qualified as a teacher of science under the Department of Science and Art. Finally,

to complete his scientific training, he studied at the Royal School of Mines for three years (1864–1867), gaining in May, 1864, the Royal Albert Scholarship. Other

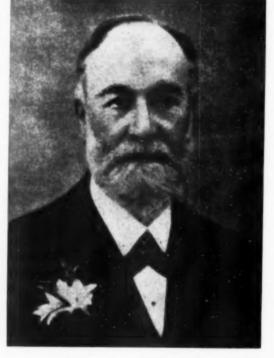
distinctions gained by Snelus were first place and gold medal for physical geography; first place and silver medal for applied mechanics; second place and bronze medal for inorganic chemistry; and finally the De la Beche medal for mining. In his second year at the Royal School of Mines, he assisted Professor Frankland at the Royal College of Chemistry, and also successfully conducted science classes at the Royal Polytechnic and other institutions, emerging in 1867 with an Associateship in Mining and Metallurgy.

He obtained his first appointment as Chemist under the Manager, Mr. Menelaus, of the Dowlais Iron and Steel Works, then the largest in the United Kingdom. At that time the rifle volunteer movement was much to the fore. Snelus had become a volunteer in 1860, receiving a commission, and was a successful shot, winning in

1864 a place in the first sixty for the Queen's Prize at Wimbledon, while in 1868 he won the first prize for small bore rifles in the Albert Competition. Other prizes fell to him, and for the twelve years 1866–1877 he competed in the volunteer match between England, Ireland and Scotland, often standing among the highest scorers.

One of the first Members of the Iron and Steel Institute, he was a frequent contributor and speaker at its meetings, his first paper being read in 1870 at Merthyr Tydfil, its subject being "The Condition of Carbon and Silicon in Iron and Steel." In the following year, he investigated the composition of the gases evolved from the Bessemer converter during the blow.

In 1871, at the Annual Meeting of the Iron and Steel Institute in London, Professor Roscoe had lectured on the spectrum analysis of the flame issuing from the



Bessemer converter. It was difficult to establish the cause of the majority of the lines in the spectrum so obtained. Various opinions were expressed that they were carbon or manganese, but Snelus considered that an analysis of the gas producing the lines would solve the problem. He collected the gas, therefore, using a long iron gas pipe, one end of which was inserted in the neck of the furnace, while to the other end glass tubes were attached at particular periods of the blow for purposes of analysis. These tubes were sealed with the blowpipe before being removed.

Samples of gas were taken every few minutes, and analysis revealed that, broadly, the carbon in the converter absorbed twice as much oxygen at one time as at another. Snelus deduced that this and the differences in the spectra were caused by varying temperatures. He also proved that the converter gas during the second half of the blow was as valuable as any gas deliberately produced in an iron works. He suggested that this gas might be collected and used as a means of saving coal for

boilers.

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American Visit

During these investigations, he was appointed a delegate of the Iron and Steel Institute for the investigation of an American puddling machine. He sailed from Liverpool early in October, visiting the Cincinnati Iron Works. His special report on the scientific features of the process exploded the theory that phosphorus was removed by liquation from the puddled ball. This discovery was the germ of the principle that was later to prove so fruitful for the steel trade. Snelus, while in the United States, examined many other puddling processes, on which he reported. He also contributed papers on the manufacture and use of spiegeleisen, and on fireclays

and other refractory materials.

In 1873, Sir William Siemens had outlined to the Iron and Steel Institute his difficulties in finding for his steel furnace a lining capable of withstanding the excessively high temperatures. Snelus stated in the discussion that in 1872 he had patented the use of limestone in place of bauxite, and had used it for lining a small Bessemer vessel with admirable results. The limestone was not raw, but ground to the plastic condition of ordinary clay, which it resembled in plasticity. Thus, it could be moulded into any form. When rammed up round a core, Thus, it could be it formed a permanent lining so long as it was in use, but if allowed to stand, it slaked away as lime. The temperature did not affect it, and it formed a hard, compact and infusible lining. Snelus suggested that Siemens would find it more infusible than bauxite, lower in cost, and probably more practical.

Elimination of Phosphorus

The experiments he thus clearly indicated were not made publicly known for seven years, whereupon they created a sensation. It became apparent that Snelus had all that time been familiar with the principle and properties of iron dephosphorization in the Bessemer converter. It is worthy of note that the basic process, both in principle and material, was publicly though incompletely pointed out to both Bessemer and Siemens by two men of considerable scientific attainment, but neither inventor utilized the valuable suggestions freely offered to them.

Snelus had long doubted the correctness of Dr. Percy's hypothesis that phosphorus was eliminated by liquation of fluid phosphide of iron from the pasty puddled ball. Examining the reaction occurring in the Dank's process, he was struck by the fact that much of the phosphorus was eliminated while the iron was fluid. Comparison of the slags in the process of Welsh refining, puddling and Bessemer converting, showed him that the slag from the puddling process, in which the phosphorus was eliminated with comparative ease, was highly basic, whereas that from the refinery, in which small amounts only of phosphorus were removed, was less basic; and that from the converter, in which none at all was removed, was highly siliceous. Other observations pointing to the same results led him to conclude that phosphorus was removed in all processes in proportion to the basic character of the slag.

At the beginning of 1872 Snelus was considering how to obtain systematically a highly basic slag in the Bessemer process. Two or three materials tried would not withstand the intense heat of the converter. It was then that he happened to observe the beneficial effects of using lime for lining the furnace. He chose and particularly specified magnesium limestone. Soon afterwards he discovered that bricks could be made of lime or limestone if the lime, when used for this purpose, were quickly crushed, compressed, and fired before it had absorbed moisture. The consolidation of the bricks called for an intense heat, especially when the lime was pure, and was facilitated by the introduction of a small quantity of

oxide of iron or other fusible base.

His first experiment was made with a few hundredweights of Cleveland pig iron melted in a cupola, then poured into the converter and blown in the usual way till the carbon lines disappeared. The first piece of steel thus produced from phosphoric iron he carefully preserved. It was free from phosphorus. This element was, however, detected in the slag. Later experiments and analyses showed beyond question that as long as the slag was kept basic, the phosphorus could be eliminated without difficulty. To make sure, he lined the same small converter with ganister, and found that the phosphorus, as previously, remained in the metal. When he came to line a 7-ton converter, however, he experienced considerable difficulty in preparing a lining sufficiently strong, and deferred further experiments while patenting his discovery.

Steelworks Management

He was often asked in later life why he did not follow up his discovery and put it into practice. The reply was that he had just taken over management of an undertaking with interests opposed to the solution of the problem. Having secured his claim by patent, he was compelled to await a more favourable opportunity of developing the invention. Moreover, Snelus remarked, there was much scepticism and no-one appeared anxious to take up the discovery. For example, none of his friends would agree to work the process.

He had confidentially told some friends of his experimental success, after it had been patented, and one, an eminent engineer, advised him not to dabble in patents as they did not pay. He added: "When you can show one thousand tons of rails made by your process, then I will believe in it." In May, 1880, this same engineer remarked that he had recently tested one thousand tons of rails made by the basic process, as compared to a thousand tons made by the old process, and found no difference. Again, a few months before Sidney Gilchrist

Thomas vaguely announced his discovery of the basic process and his application of it, Snelus gave a short account of its principal features to the Iron and Steel Institute.

The rival inventors sensibly avoided litigation. Thomas and Gilchrist, who had worked out a method of making the process a practical success, agreed with Snelus to submit their claims for a division of the profits to the judgment of Sir William Thomson. The patent rights of Snelus extended to America and the United Kingdom, but not to the continent. The American patent had been taken out by Snelus on the recommendation of his friend Cooper, of Cooper and Hewitt, New York, but not until 1878, which was almost too late. Snelus had delayed until he had perfected all details.

When Thomas and Gilchrist had established their process, Snelus continued to study it from the practical point of view, being convinced that the elements found in combination with iron were eliminated in a basic-lined furnace in the order: silicon, carbon, phosphorus, manganese, sulphur. In the acid converter the order was: silicon, carbon, manganese; no phosphorus or sulphur being removed at all. He believed also that the process would produce from the poorest ores a steel as good as Bessemer steel, and also the highest quality of cutlery steel. In his view, a cutting steel of cutlery type should be free from silicon, have the right proportion of carbon, and be absolutely free from phosphorus.

The works whose management had prevented Snelus from developing his discovery was the West Cumberland Steel Works, of whose Bessemer Department he became the Manager soon after taking out his British patent. A few months later he was promoted to General Manager of the entire works. Under him it flourished, and his scientific and practical knowledge of the Bessemer process enabled him to introduce valuable improvements.

When he was in America he had studied the advantages of the Holley patent for Bessemer vessel bottoms, and made a special journey of 1,000 miles from New York to Harrisburg to convince himself that he had thoroughly grasped the invention, which he later introduced to the West Cumberland Works, where it continued in use. In the following year he visited Germany, where he saw the process of ascensional casting, under Mr. Pink at Hoerde. This improvement also he would have introduced at West Cumberland, but the size and construction of the Bessemer pits there were not suitable.

Blast Furnace Iron for Converters

Snelus was among the first in England to advocate the conversion of fluid iron direct from the blast furnace into steel, as Bessemer had proposed. At the West Cumberland Iron and Steel Works the distance between blast furnace and converters was 350 yards. To carry the molten metal he designed, therefore, a large ladle of 8 tons capacity, so placed in the centre of a carriage that no balance weight was needed, and a simple cast steel worm and wheel would turn it over. The weight of the entire apparatus was below 10 tons. In practice, about 3½ tons of molten iron was tapped from each of two furnaces into the ladle, to ensure a uniform charge. Tapping of both furnaces often took only five minutes, and in less than that time the charge was weighed, taken to the converter and poured into it. One ladle lasted from 100 to 200 casts before the "skull" needed to be removed. A better yield of metal was obtained and a

saving of labour and fuel estimated at 4s. to 5s. per ton achieved. The steel was also of better quality.

Snelus was one of the first steel makers in England to adopt the soaking pit, invented by Gjers, and at The Iron and Steel Institute meeting at Vienna in September, 1882, he spoke strongly in its favour and gave practical advice on its construction. At the Paris Exhibition of 1878 he exhibited, on his own behalf, an elaborate collection of analysed samples illustrating the manufacture of iron and steel in England, and was awarded a gold medal. The collection was bought by Professor Deure for the Polytechnic School at Aix-la-Chapelle.

The President of the Iron and Steel Institute in 1883, Mr. B. Samuelson, M.P., presented the Bessemer Gold Medal to Snelus, as the first man to make impure iron into pure steel in a Bessemer converter lined with basic materials. In his reply, Snelus gave every possible meed of credit to his early teachers and to other discoverers.

Elected a Fellow of the Royal Society in 1887, he died at his residence, Ennerdale Hall, Frizington, Cumberland, on June 18th, 1906, at the age of 69.

Oxygen in Steelmaking

(Continued from page 1)

time expected to create an enormous demand for tonnage

oxygen, has proved disappointing.

For some time now, the use of oxygen for rapid decarburisation in the electric arc furnace has been established practice in this country, the net effect being a saving of both time and electric power. The oxygen lance is desirable in the production of low carbon steels, but it is essential if charges containing a high proportion of stainless steel scrap are to be refined. Charges of 100% stainless steel scrap can be refined to below 0.06% carbon with a chromium recovery of over 90%, because the heat generated in the early stages of the blow by the oxidation of a small proportion of the chromium present raises the bath temperature to more than 1.800 °C., in which region oxidation of carbon is preferential to that of chromium.

In the end, decisions regarding the use of oxygen in a particular process depend on economic factors, and whilst certain processes can be operated extremely successfully from this point of view with delivered liquid supplies, others depend on the availability of large-scale supplies at lower cost from tonnage oxygen plants situated in or near the works itself. This explains why the use of oxygen lancing in electric arc furnaces is now established practice in this country, whilst we lag behind Continental operators in other respects. The converter steelmaker uses sufficient oxygen to justify the installation of a tonnage oxygen generator, and thus has oxygen available for secondary purposes such as desiliconisation, etc., at tonnage prices. This state of affairs in the United Kingdom is now being rectified by the erection in steelmaking areas of centrally-disposed tonnage oxygen plants, from which the gas can be distributed by pipeline to neighbouring steelworks. With these developments, progress in the application of oxygen in the U.K. iron and steel industry should become increasingly rapid.

Change of Telephone Number

British Insulated Callender's Cables, Ltd., announce that the telephone number of their Swansea office has been changed to Swansea 52094.

The Effect of Surface Finish on Fatigue

By B. Cina, B.Sc., Ph.D.

Brown-Firth Research Laboratories, Sheffield.

Emery polishing of a wide range of alloy steel fatigue test pieces gives artificially high fatigue strength values due to the cold working of the test piece surface. Truer fatigue strength values are obtained by electropolishing the test pieces, which removes the cold worked layer, reduces the scatter of fatigue lives, and largely resolves the difference in fatigue strength between rotating bending and direct stress fatigue tests. Stress relieving after mechanical polishing gave similar results to electropolishing.

THE great majority of fatigue tests are carried out on samples whose surfaces have been finally prepared for testing by polishing with emery paper, the final direction of polishing being either circumferential or longitudinal. In many cases, for example, for rotating bending test pieces, successively finer grades of emery paper are all manually applied to the test piece in a circumferential direction. This is done because of the ease of the method. However, consideration of this procedure suggested that it might give erroneous results. It was known from metallographic practice that the scratches left by one grade of emery paper are more easily removed by the next finer grade if the direction of polishing is turned through 90°. Repeated circumferential polishing might, therefore, be expected inefficiently to remove all previous scratches at any stage in polishing. The notch effect of such hidden scratches may well tend to reduce fatigue strength. On the other hand, polishing with emery paper may introduce cold work effects into the surface, the effect of which may be to increase the While these effects of scratches and fatigue strength.

cold work may tend to cancel one another, the relative magnitude of each will vary with the sample, the material, and the operator. They were considered worth investigating, since they might affect the mode of initiation of fatigue failure, and also contribute, in part, to the well-known scatter of fatigue results.

Experimental Details

Materials Used

In order to determine the generality of the aforementioned effects, the work was carried out on a fairly wide range of steels, though mainly of the stainless variety. These included stable and unstable austenitic, fully ferritic (δ), and martensitic stainless steels, and a low alloy steel. The analyses of the several steels are given in Table I.

All the materials, except Code No. 80714, were received in the form of hot rolled bar, and were heat treated in this condition prior to testing. All the bars were 3 in. diameter, except Code Nos. 08835 and 65937, which were 11 in. diameter. Code No. 80714 was received

TABLE L-ANALYSES OF ALLOY STEELS STUDIED

		Actual Composition								
Rominal Percentage Composition	Code No.	C %	84 9/ 0	Mn %	8 %	P %	Cr %	Ni %	N %	
13/0-2 Cr-C 13/0-2 Cr-C 13/0-2 Cr-C 20/0-04 Cr-C 21/0-06 Cr-C 21/0-06 Cr-C 18/8 Cr-Ni 24/22 Cr-Ni 9/3/8/0-6 Mn-Cr-Ni-C 9/3/8/0-6 Mn-Cr-Ni-C	HJ1304 HJ1124 HJ4176 08835 01875 01876 XF75751 08995 80714 69190 65937	0·21 0·21 0·24 0·037 0·06 0·06 0·08 0·13 0·65 0·63 0·32	0-47 0-35 0-23 0-63 0-25 0-25 0-64 1-23 0-35 0-41 0-27	0.53 0.47 0.35 0.70 0.73 0.77 0.86 7.16 7.45 0.61	0·013 0·011 0·027 0·028 0·017 0·017 0·017 0·006 0·006 0·008	0·026 0·020 0·021 0·018 0·015 0·016 0·016 0·019 0·034 0·014	13·08 13·28 13·06 21·42 20·68 20·76 18·24 23·64 3·84 4·00 0·08	0·29 0·41 0·35 0·17 0·21 0·21 8·75 22·00 8·45 8·35 8·35	0 · 025 0 · 032 0 · 025 0 · 015 0 · 027 0 · 025 0 · 049 0 · 041 0 · 018 0 · 015	

TABLE II .- MECHANICAL PROPERTIES OF ALLOY STEELS STUDIED.

Nominal Percentage Composition Code No.	Code No. Heat Treatment®	L.P.†	P.S.* (tons/sq. in.)				U.T.S.	EL.	R.A.	
			sq. in.)	0.05%	0.10%	0-20%	0.50%	sq. in.)	%	%
(3/0·2 Cr-C (3/0·2 Cr-C (3/0·2 Cr-C (3/0·2 Cr-C (3/0·2 Cr-C (3/0·2 Cr-C (1/0·06 Cr-C (1/0·06 Cr-C (1/0·06 Cr-C (8/3 Cr-Ni (4/22 Cr-Ni (4/22 Cr-Ni (4/28 Cr-Ni-C)	HJ1304 HJ1304 HJ1124 HJ4176 08835 01875 01876 XF75751 08995 80714 69190	$ \begin{array}{l} & hr,/1,000^{\circ} \ C,/O,Q, \\ & hr,/1,000^{\circ} \ C,/O,Q, \ + \ 1 \ hr,/650^{\circ} \ C,/W,Q, \\ & hr,/1,000^{\circ} \ C,/O,Q, \ + \ 1 \ hr,/650^{\circ} \ C,W,Q, \\ & hr,/1,000^{\circ} \ C,/O,Q, \ + \ 1 \ hr,/650^{\circ} \ C,W,Q, \\ & 2 \ hr,/800^{\circ} \ C,/W,Q, \\ & 2 \ hr,/800^{\circ} \ C,/W,Q, \\ & 1 \ hr,/1,050^{\circ} \ C,/W,Q, \\ & hr,/1,050^{\circ} \ C,/W,Q, \\ & hr,/1,050^{\circ} \ C,/A,C, \\ & hr,/1,050^{\circ} \ C,/A,C, \\ \end{array} $	40·3 35·0 23·5 27·8 15·0 14·6 10·0 7·9 5·7 11·7 15·6	62·4 42·2 37·4 39·9 18·8 20·5 18·1 15·7 15·6 19·8 20·1 Upper Y.P.	71.0 43.5 40.0 43.6 19.4 21.4 18.9 16.7 17.4 20.4 20.6 Lower Y.P.	81·0 45·1 42·0 44·6 19·9 91·9 11·8 17·8 18·8 20·9 21·4	92.6 46.6 43.8 45.6 90.0 22.1 19.9 18.9 20.6 21.7 22.1	114·0 56·4 54·5 57·2 28·6 31·4 30·7 42·3 42·0 50·5 50·6	16·5 24 25·5 34 42 41 43·5 65 50 82·5 81	45 62 64 65 71 73 76 73 66 71 - 5
/0-3 Ni-C	65937	hr./850° C./B.Q. + 1 hr./650° C./A.C. + 1 hr./650° C./F.C.	38-0	44.0	41.6			49.0	31.5	69

⁶ In this paper briefly defined by the time at temperature and the subsequent method of cooling. Thus, ½ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q., indicates oil quenching after heating at 1,000° C. for half an hour, followed by tempering for an hour at 650° C. and water quenching. The significance of the abbreviations is as follows: W.Q.—water quench; O.Q.—oil quench; B.Q.—brine quench; A.C.—air cool; and F.C.—furnace cool. ↑ Limit of Proportionality. ‡ Proof Stress.

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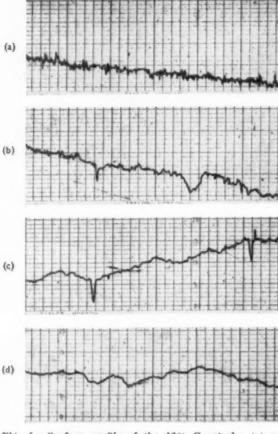


Fig. 1.—Surface profile of the 13% Cr steel: (a) as mechanically polished; (b) as (a) \pm 15 seconds electropolishing; (c) as (a) \pm 45 seconds electropolishing; (d) as (a) \pm 5 minutes electropolishing.

as $\frac{\epsilon}{8}$ in. diameter cold drawn bar, but was re-softened prior to testing. The mechanical properties of the alloy steels in their several heat treated conditions are given in Table II.

Preparation of Fatigue Test Pieces

Heat treated bar lengths, each suitable for the preparation of one test piece, were machined in the normal manner. The final test piece, of Wöhler type (rotating bending, single point loading), had a minimum diameter of 0.250 in. and a continuous radius in the test portion of 2.75 in. Its overall length was 3.75 in. A few tests were also carried out on similar type test pieces, of 0.160 in. minimum diameter, 1.5 in. continuous radius, and 2.5 in. overall length. Samples to end with a mechanically polished surface were polished with successively finer grades of emery paper, ending with 000 quality, all in a circumferential direction. Polishing in this manner removed about 0.004 in. depth of material, as measured at the minimum diameter of the test piece. Stainless alloy steels to be electrolytically polished were first emery polished as far as 1M quality, all in a circumferential direction, and then electrolytically polished for 5 minutes in a bath containing 20: 1 glacial acetic acid: perchloric acid as electrolyte, at an applied voltage of

60–65, and using a stainless steel sheet as cathode. The low alloy steel was polished in the same bath, but at an applied voltage of about 35, and for $10-12\frac{1}{2}$ minutes. These electropolishing treatments removed about $0\cdot002-0\cdot003$ in. of material from the minimum diameter of the test piece. The latter was measured to $\pm~0\cdot0001$ in., both prior to and half way through the electropolishing of each sample. This kept a check on the rate of electropolishing, and avoided applying a micrometer to the test piece surface after final electropolishing.

Determination of S-N Curves

Most of the tests, except for those of 0·160 in. minimum diameter, were carried out on two Avery Wöhler machines operating at 2,900 cycles/minute and using samples of 0·250 in. minimum diameter. The 0·160 in. diameter test pieces were fatigued at 4,750 cycles/minute. All the tests on any one alloy were carried out on the same machine. In general, duplicate tests were run at each stress level examined. The fatigue limit was generally based on two samples remaining unbroken for 25×10^6 cycles.

Preliminary Experiments

Nature of a Mechanically Polished Surface

To determine whether repeated circumferential polishing with emery paper did, in fact, cause lapping over of coarse scratches by the finer grades of paper, the surface of a 0.250 in. diameter sample of a 13% Cr steel in the hardened and 650° C. tempered condition was examined by means of a Talysurf surface measuring instrument. The latter automatically records the actual surface profile at vertical and horizontal magnifications of \times 20,000 and \times 100 respectively. The sample was mechanically polished circumferentially as far as 000 emery paper. A trace was then taken of the surface in the longitudinal direction, i.e., at right angles to the scratches. The sample was electropolished for various periods from 15 seconds up to 5 minutes, and examined intermittently with the Talysurf instrument. The results prior to and after 15 seconds, 45 seconds, and 5 minutes, are illustrated in Figs. 1a-1d, respectively. Figs. 1b and 1c clearly reveal that scratches much deeper than those indicated in Fig. 1a are present beneath the apparent surface of the latter. In this way, it has been found that such deep scratches can be present up to about 0.0005 in. beneath the original 000 emery polished surface. Electropolishing for 5 minutes removed a depth of about 0.001 in. of this material. Similar results were obtained for a 4% Ni-Cr-Mo steel. These experiments confirmed the lapping over and incomplete removal of coarse scratches by finer grades of paper in circumferential polishing, and established that at least 0.0005 in. material had to be removed by electrolytic polishing. The standard technique adopted removed between 0.001 in. and 0.0015 in. depth of material.

Effect of the Extent of Mechanical Polishing Prior to Electropolishing

Since the final grades of emery paper had a lapping effect, fatigue test pieces to be electropolished were, in fact, first mechanically polished only as far as IM emery, since more material was removed by the electropolishing than by the final 0 to 000 grades of emery paper. The same fatigue results were obtained by electropolishing after a final IM emery as after a 000 emery finish. Thus, the fatigue lives detailed in Table III were obtained on a

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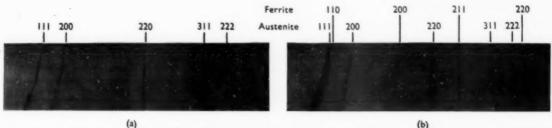


Fig. 2.—X-ray diffraction photographs of the 18/8 Cr-Ni steel: (a) as electropolished; (b) as mechanically polished.

13% Cr steel, Code No. HJ4176, in the $\frac{1}{2}$ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q. condition, tested at \pm 34 tons/sq. in. In addition, no significant difference in surface profile was observed after electropolishing for the two prior modes of emery polishing.

Effect of Time of Electropolishing on Fatigue Life

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The fatigue limits of some $9/3/8/0\cdot6$ Mn-Cr-Ni-C steel test pieces (of Code No. 80714 and in the $\frac{1}{2}$ hr./1,050° C./A.C. condition), after electropolishing for $2\frac{1}{2}$, 5 and 15 minutes to remove $0\cdot0008$ in., $0\cdot0018$ in., and $0\cdot0056$ in. from the diameter, were \pm 20, \pm 22, and \pm 22 tons/sq. in., respectively, i.e., a constant value was obtained only after 5 minutes electropolishing. The lower fatigue limit can be explained by a depth of $0\cdot0004$ in. of material removed by electropolishing being inadequate completely to clear the 1M finish of lapped-over coarser emery polishing scratches.

There was no significant difference in fatigue life at the stresses examined for test pieces of the 13% Cr steel, Code No. HJ1304, which had been electropolished to remove 0.001 in. or 0.002 in. of material from the diameter. Similar results were also obtained for samples of the 18/8 Cr-Ni steel, Code No. XF75751, electropolished to remove 0.001 in. or 0.005 in. from the diameter.

X-ray Diffraction Examination as a Check on Effectiveness of Electropolishing

As it was important to remove not only scratches, but also cold work effects, test pieces of 18/8 Cr-Ni steel, Code No. XF75751, in the 1 hr./1,050° C./W.Q. condition, were examined by X-ray diffraction glancing-angle technique, both as 000 emery polished and as electropolished for 5 minutes. This alloy was chosen because of its great sensitivity to cold work. The X-ray diffraction photographs are shown in Figs. 2a and 2b for the electropolished and mechanically polished samples, respectively. While Fig. 2a indicates a stress-free fully austenitic structure, Fig. 2b shows that the mechanical polishing has not only left the austenite crystals in a fragmented micro-stressed condition, but has also caused about 25% of this material to transform to ferrite. This is a martensitic transformation induced by cold work,1 and, together with the appearance of the austenite diffraction

TABLE III.—EFFECT OF MECHANICAL POLISH, PRIOR TO BLECTRO-POLISH, ON FATIGUE LIFE.

Method of Surface Preparation	Fatigue Life at \pm 34 tons/sq, in. (cycles)
1M Emery Polish + 5 Minutes Electropolish 000 Emery Polish + 5 Minutes	213,300 ; 273,000 ; 265,200
Electropolish	302,400; 260,100; 224,500

lines, is clear evidence that the austenite has been fairly heavily plastically deformed, i.e., a mechanically polished surface is one left in a cold worked condition. This X-ray work also confirmed that 0·001 in. depth of material removed by the electropolishing was adequate to get rid of the cold work effects of prior mechanical polishing. Hempel² has also found that the mechanically polished surface of a Cr-V steel fatigue test piece is in a cold worked condition, compressive stresses of 25-38 tons/sq. in. being detected to a depth of 0·0008-0·002 in.

Results

Effect of Electropolishing on the Fatigue Life of Wöhlertype Test Pieces

This was investigated on six different types of steel. Five of these were tested on two machines employing $0\cdot250$ in. minimum diameter test pieces. These steels comprised the following: $13\,\%$ Cr, $20\,\%$ Cr, 25/20 Cr-Ni, $9/3/8/0\cdot6$ Mn-Cr-Ni-C, and 18/8 Cr-Ni. The sixth was the plain $3\,\%$ Ni steel, and was tested on a machine employing a $0\cdot160$ in. minimum diameter test piece. The results were as follows:

13% Cr Steel.—This steel was tested in two conditions of heat treatment; (a) after $\frac{1}{2}$ hr./1,000° C./O.Q., representing a fully martensitic condition; and (b) after $\frac{1}{2}$ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q., representing a tempered martensite. The results for material of Code No. HJ1304 are shown in Figs. 3a and 3b, for the (a) and (b) conditions, respectively. For the fully martensitic condition, electropolishing has lowered the fatigue limit from \pm 50 to \pm 46·5 tons/sq. in., and has also markedly reduced the fatigue life at higher stresses. The reproducibility of results at the higher stresses clearly shows the difference in life for the two methods of surface preparation to be significant.

For the tempered material in Fig. 3b, the fatigue limit has again been lowered by electropolishing, this time from \pm 35 to \pm 29 tons /sq. in. At higher stresses, fatigue life has also been reduced, but to a lesser extent than for fully martensitic material.

This work on a tempered 13% Cr steel was repeated on samples from a second cast, Code No. HJ1124, and the results obtained confirmed those in Fig. 3b. The fatigue limit of the second cast was reduced from \pm 33 to \pm 27·5 tons/sq. in. by electropolishing, showing that the effect was not due primarily to the particular cast chosen

20% Cr Steel.—Samples of Code No. 01875 were tested after a heat treatment of 2 hr./800° C./W.Q., which left the material in a fully softened condition, the microstructure being simple polygonal δ -ferrite grains. The S-N curves for this alloy steel are shown in Fig. 4.

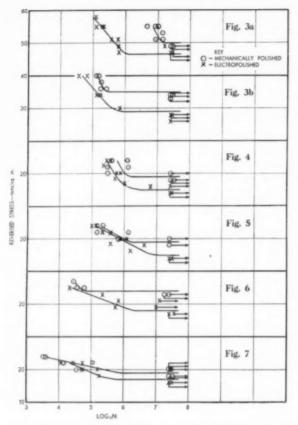


Fig. 3.—Wohler S-N curves for the 13% Cr steel, Code No. HJ1304 : (a) after $\frac{1}{2}$ hr./1,000° C./O.Q. ; (b) after $\frac{1}{2}$ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q.

Fig. 4.—Wohler S-N curves for the 20% Cr steel, Code No. 01875, after 2 hr./800° C./W.Q.

Fig. 5.—Wohler S-N curves for the 25/20 Cr-Ni steel, Code No. 08995, after 1 hr./1,050° C./W.Q.

Fig. 6.—Wohler S-N curves for the 9/3/8/0·6 Mn-Cr-Ni-C steel, Code No. 69190, after ½ hr./1,050° C./A.C.

Fig. 7.—Wohler S-N curves for the 18/8 Cr-Ni steel, Code No. XF75751, after 1 hr./1,050° C./W.Q.

Electropolishing, which removed about 0.003 in. material from the minimum diameter, has lowered the fatigue limit from \pm 19 to \pm 15 tons/sq. in., and also seems to have reduced fatigue life at higher stresses, although the scatter of results for the mechanically polished samples tends to make the difference less apparent.

25/20~Cr-Ni~Steel.—Samples of this stable austenitic alloy steel, Code No. 08995, were treated for $\frac{1}{2}$ hr./ 1.050° C./W.Q., which left the material in a fully softened condition, the microstructure consisting of simple polygonal austenite grains. Electropolishing, which removed about 0.004 in. from the minimum diameter of the test piece, lowered the fatigue limit from \pm 19 to \pm 15 tons/sq. in., while the effect on fatigue life was possibly to reduce it, although scatter of results made this less certain to determine. These results are shown in Fig. 5.

 $9/3/8/0 \cdot 6$ Mn-Cr-Ni-C Steel.—This also was a stable austenitic alloy steel material of Code No. 69190, being softened by a treatment of $\frac{1}{2}$ hr./1,050° C./A.C., which resulted in a microstructure of simple polygonal austenite grains. The S-N curves for this alloy are shown in Fig. 6. Electropolishing, which removed about $0 \cdot 003$ in. from the minimum diameter, has resulted in a decrease in fatigue limit from \pm 24 to \pm 18 tons/sq. in. The near absence of a surface finish effect at higher stresses will be discussed later.

18/8~Cr.Ni~Steel.—The material used for this type of alloy steel, from Code No. XF75751, was given a softening treatment of 1 hr./1,050° C./W.Q. and had a structure of unstable austenite, i.e., martensitic transformation of the austenite to ferrite could be induced by plastic deformation. Its S-N curves are shown in Fig. 7. Electropolishing has lowered the fatigue limit from \pm 19 to \pm 17 tons/sq. in. The scatter at higher stress values for both modes of surface finish makes it difficult to determine the precise effect of electropolishing at these stress levels.

3% Ni Steel.—The samples of this alloy steel, Code No. 65937, were tested in a double tempered condition, namely, $\frac{1}{2}$ hr./850° C./B.Q. + 1 hr./650° C./A.C. + 1 hr./650° C./F.C. The microstructure was, of course, tempered martensite. The fatigue results are illustrated in Fig. 8. Electropolishing has reduced the fatigue limit from \pm 29 tons/sq. in. to \pm 24½ tons/sq. in., while the fatigue life at higher stresses seems to be little affected by the electropolishing.

Effect of Electropolishing on the Fatigue Life of Samples Tested in Direct Stress

Haigh fatigue test pieces, with a gauge length approximately $0 \cdot 178$ in. diameter $\times \frac{1}{4}$ in. long, were mechanically and electrolytically polished as were the Wöhler test pieces, and were tested in reversed stress and also in pulsating tension at 6,000 cycles per minute. The results were as follows:—

Reversed Direct Stress.—This work was done on a 20% Cr steel, Code No. 08835, after a softening treatment of $2 \text{ hr./800}^{\circ}$ C./W.Q. The S-N curves are shown in Fig. 9. Electropolishing, which removed about $0 \cdot 003$ in. from the gauge diameter, has only lowered the fatigue limit from $\pm 15 \cdot 5$ to ± 15 tons/sq. in., and at higher stresses also caused some reduction in fatigue life, although the results suggested that this effect would decrease with increasing stress amplitude.

Pulsating Tension.—Only a few tests were carried out by this method of fatigue testing, and these were done on a 20% Cr steel, Code No. 01876, after a softening treatment of 2 hr./800° C./W.Q. The results suggested that the fatigue limit was not affected by the surface finish

Effect of Stress Relief Treatment on the Fatigue Strength of Mechanically Polished Test Pieces

Consideration of all the foregoing results suggested that the longer fatigue life and higher fatigue limit of samples mechanically, as compared with electrolytically, polished might be due to cold work effects introduced to the surface in the one case by the mechanical polishing. The effect of this cold work would have been to strengthen the surface and to introduce surface compressive stresses²⁻⁴ which would be expected to increase fatigue strength. If this hypothesis were correct, a stress relief treatment given to a Wöhler fatigue test piece after

mechanical polishing should give a shorter fatigue life and a lower fatigue limit than one tested as mechanically polished. To test this hypothesis, two series of samples of 3% Ni steel, Code No. 65937, were identically hardened and tempered. One series was then kept as unmachined blanks while the other was machined to Wöhler test pieces and mechanically polished as far as 000 emery paper to 0·160 in. minimum diameter. The blanks and the polished test pieces were then given an identical stress relieving treatment as follows:—

Heated to 650° C. in 3 hours, held 1 hour at 650° C. and furnace cooled to room temperature in about 4 hours. The polished test pieces were given this treatment in vacuo, and were perfectly bright at the end of the cycle. The blanks were then machined and polished as far as 000 emery paper. Both series of test pieces, which had thus had identical heat treatments, save that one had been stress relieved after and one before mechanical polishing, were tested on the same machine and the results are shown in Fig. 8. The fatigue limit has decreased from \pm 29 to \pm 24.5 tons/sq. in. for the series stress relieved prior to and after mechanical polishing, respectively, in line with prediction. The decrease in fatigue limit due to stress relief is the same as that due to electropolishing. Strictly speaking, if stress relief is complete, it should give a slightly lower fatigue limit than electropolishing, since the former still leaves emery polishing scratches on the surface which should act as notches and lower the fatigue limit. That such a lower fatigue limit was not obtained in the present case is possibly due to the stress relief treatment being incomplete, or to the notch effect being too small to detect at the lower stresses.

A few similar tests were carried out on a 13% Cr steel, Code No. HJ4176. The stress relief cycle for this material was similar to that for the 3% Ni steel. The previous heat treatment had consisted of ½ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q. Fatigue lives were determined at only two stress levels, the results being shown in Fig. 10. Stress relief seems to have caused a significant reduction in fatigue life at both stresses, in line with

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Table IV gives the mechanical properties of this steel, both prior to and after stress relief, and shows no significant change in proof stress, tensile strength or ductility. The increase in limit of proportionality is probably due to the slower rate of cooling from the stress relieving temperature. All this, therefore, implies that there could have been no significant microstructural change in the main body of the material as a result of the stress relief treatment, and that the effect of the latter was simply as its name implies.

Vacuum stress relief treatment of 2 hr./500° C., carried out by Hempel² on mechanically polished Cr-V and plain C steel fatigue test pieces also caused a reduction in fatigue limit, although to a lesser extent than that caused by electropolishing. This was possibly due to the temperature employed being too low for full stress relief.

So far as the actual surface of the specimen is concerned, the diamond pyramid hardness figures for the

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Fig. 12

Fig. 12

Fig. 12

KEY

O MECHANICALLY POLISHED

A COLD DIAWN 20% AND ELECTROPOLISHED

W COLD STREETORD 20% AND ELECTROPOLISHED

O COLD STREETORD 20% AND ELECTROPOLISHED

O COLD STREETORD 20% AND ELECTROPOLISHED

TO COLD STREETORD 20% AND ELECTROPOLISHED

Fig. 8.—Wohler S-N curves for the 3% Ni steel, Code No. 65937, after $\frac{1}{2}$ hr./850° C./B.Q. + 1 hr./650° C./A.C. + 1 hr./650° C./F.C.

Fig. 9.—Haigh S-N curves for the 20% Cr steel, Code No. 08835, after 2 hr./800° C./W.Q.

Fig. 10.—Wohler S-N curves for the 13% Cr. steel, Code No. HJ4176, after $\frac{1}{2}$ hr./1,000° C./O.Q. + 1 hr./650° C./ W.Q. + 1 hr./650° C./F.C.

Fig. 11.—Wohler S-N curves for the 9/3/8/0.6 Mn-Cr-Ni-C steel, Code No. 69190, after $\frac{1}{4}$ hr./1,050° C./A.C. + 20% reduction by cold drawing.

Fig. 12.—As Fig. 11, but 20% reduction obtained by cold stretching instead of cold drawing.

surface of a stress relieved 13% Cr steel test piece and for the surface of a sample stress relieved before polishing were 277 and 279, respectively. These results were obtained with a 1 kg. load. The depth of penetration of the indenter for hardnesses of this order is only 0.00065 in. It follows, therefore, that any structural change induced by the polishing and subsequent stress relief would have been confined to a zone of material much less than 0.00065 in. deep, since at the latter depth no such effect was detected.

Effect of Cold Work on Fatigue

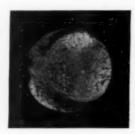
While the role of residual stresses in fatigue has been fairly well established, conflicting results have been found for the effect of cold work. In the light of the foregoing section, it was important to know whether cold work could affect fatigue strength. Accordingly, some of the 9/3/8/0.6 Mn-Cr-Ni-C steel in the $\frac{1}{2}$ hr./

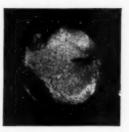
TABLE IV.—MECHANICAL PROPERTIES OF A 13% CR STEEL PRIOR TO AND AFTER STRESS RELIEF TREATMENT.

Condition	L.P.		P.S. (tor	U.T.S.	El.	R.A.		
Condition	tons/sq. in.	0.05%	0.1%	0.20	0.5%	tons/ sq.in.		%
Stress Relief	27.8	39-9	42.6	44-6	45.6	57-2	24	65
Relief	34-4	41-6	43-0	44-5	46-4	56-0	24	63









(a) (b) (c) (d) Fig. 13.—Wohler fatigue fractures of the 13% Cr steel, Code No. HJ1304, after $\frac{1}{2}$ hr./1,000° C./O.Q. + 1 hr./650° C./W.Q.: (a), (b) and (c), electropolished and tested at \pm 40, \pm 34 and \pm 30 tons/sq. in. respectively; (d) mechanically polished and tested at \pm 40 tons/sq. in.

 $1,\!050^\circ$ C./A.C. softened condition was uniformly cold drawn 20% and some was stretched 20% to give the mechanical properties in Table V.

The fatigue limit for both methods of cold working was then determined on a Wöhler machine, using 0·250 in. minimum diameter test pieces which had been electropolished prior to testing. This method of preparation was adopted to avoid spurious surface effects believed to be introduced by mechanical polishing as described previously. The results are shown in Figs. 11 and 12. The S-N curve for as-softened material is included in each of these sets of curves for comparison. Cold work has clearly resulted in an increase in the fatigue limit of 8 and 9 tons/sq. in. for drawing and stretching, respectively. This should be compared with the increase of 6 tons/sq. in. obtained solely by surface cold working (mechanical polishing) as shown in Fig. 6. There is, therefore, a degree of compatibility in all these results.

Fatigue Fractures of Mechanically and Electropolished Wöhler Test Pieces

There was generally a characteristic appearance of the fractures of test pieces polished in these two ways. Mechanically polished test pieces tended to crack at only one or two points, and to fail by the propagation of one

TABLE V.—MECHANICAL PROPERTIES OF A 9/3/8/0·6 Mn-Cr-Ni-C STREL AFTER COLD WORK.

Details of Cold tonn/sq. in.		P.S. (tons/sq. in.)				U.T.8.	El.	R.A.
	0.05%	0.10%	0.20%	0.50%	tom/sq. in.	%	0,0	
Nil (As softened) Drawn	15-6	20-1	20-6	21.4	22-1	50-6	81	66-3
20% Stretched	16-0	44.0	51.0	55-7	57 - 7	67.3	42.5	57-3
20%	12-0	42-4	48-3	49-2	50-0	61-7	55	61-5

crack. Cracking in electropolished samples tended to commence at very many points on the surface, and many of these had propagated radially well into the section before final failure. These statements now require some qualification. In one of the alloy steels, mechanically polished test pieces could also show multi-origin cracking at stresses sufficiently high above the fatigue limit, while for all the alloy steels, electropolished test pieces showed decreasing numbers of cracks as the applied range of stress approached the fatigue limit. Thus, Figs. 13a, 13b and 13c show a decreasing number of cracks in the fractures of electropolished Wöhler test pieces of 13% Cr steel, Code No. HJ1304, tested in the hardened and tempered condition at \pm 40, \pm 34 and \pm 30 tons/sq. in., respectively. The fatigue limit of this alloy steel was ± 29 tons/sq. in. (see Fig. 3b). For comparison, Fig. 13d shows the fracture of the corresponding mechanically polished sample tested at ± 40 tons/sq. in. Not more than one or two cracks are visible. Thus, for the same stress level, or at a stress level higher than the appropriate fatigue limit by a fixed amount, cracking commenced at a much smaller number of points for test pieces mechanically polished than for those electrolytically polished. The 9/3/8/0.6 Mn-Cr-Ni-C steel showed multi-origin cracking in the fractures of both mechanically and electrolytically polished test pieces. However, the fatigue results for the steel were somewhat anomalous, and will be dealt with in the Discussion. In addition to the many cracks observed on the actual fracture surface of electropolished test pieces, corresponding numbers of circumferential transcrystalline cracks were observed on the radiused portion of the test pieces adjacent to the main fracture, and up to distances of about in from the latter. The majority of these cracks were of the order of only one grain diameter, but very much longer ones were also found. For mechanically

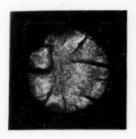




Fig. 14.—(a) and (b) as Figs. 13a and 13d; cracking adjacent to main fracture.

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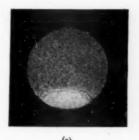
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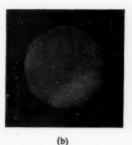


Fig. 15.—Wohler fatigue fractures of the 25/20 Cr-Ni steel, Code No. 08995, after $\frac{1}{2}$ hr./1,050° C./W.Q.: (a) as electropolished; (b) as mechanically polished. Both were tested at \pm 24 tons/sq. in.

Fig. 17.—Wohler fatigue fractures of the 13% Cr steel, Code No. HJ1304 after ½ hr./1,000° C./O.Q.: (a) as electropolished; (b) as mechanically polished. Both were tested at ± 55 tons/sq. in.

polished test pieces, few such cracks were ever found. Figs. 14a and 14b illustrate this cracking in the radius for electro and mechanically polished test pieces, respectively. In Fig. 14b, the crack tends to follow the emery scratches. Similar fracture and cracking characteristics were observed for the 3% Ni, 20% Cr, 18/8 Cr-Ni and 24/22 Cr-Ni steels. Fractures of the last named steel are illustrated in Figs. 15a and 15b. They should be compared with Figs. 13a and 13d. The three series of 3% Ni steel test pieces, namely (a) electropolished, (b) mechanically polished, and (c) mechanically polished and stress relieved, afforded instructive sets of fractures: (b) showed single origin fractures at all stresses investigated, while (a) and (c) showed multi-origin fractures, but only at the higher stresses. These features are illustrated in Figs. 16a, 16b and 16c for samples all tested at \pm 30 tons/sq. in. The multi-origin fractures in the stress relieved test pieces are in keeping with the notch effect of the polishing scratches as previously described. Similar multi-origin fractures have been observed by others2.5.6 in various electropolished low alloy steel test pieces fatigued at stresses well above the fatigue limit, and also in mechanically polished test pieces having some stress concentration characteristic such as a groove or sharp fillet.7

Finally, the fracture of the 13% Cr steel in the fully hardened condition ($\frac{1}{2}$ hr./1,000° C./O.Q.) should be described. Despite the marked difference in fatigue life for the two methods of surface preparation, as shown in Fig. 3a, there was no significant difference in the appearance of the corresponding fatigue fractures. These are illustrated in Figs. 17a and 17b for an applied stress range of \pm 55 tons/sq. in. In both cases, cracking commenced at only one point and developed to give the common thumb-nail type of fatigue fracture. Even at \pm 58

tons/sq. in., 11½ tons/sq. in. above the fatigue limit, an electropolished test piece still showed only single origin failure.

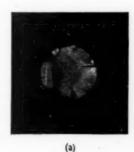
Thus, the results in this section can be summed up as follows: with the exception of the as-hardened 13% Cr steel, fatigue cracking of electropolished and mechanically polished test pieces tended to commence, respectively, at many points round and near the minimum diameter and at only one or two such points. This difference became less significant as the applied stress range approached the respective fatigue limits for both methods of surface preparation.

Apparent Discrepancy between Haigh and Wöhler Fatigue Limits

It is well known that a lower fatigue limit is obtained by testing in fully reversed direct stress than in rotating bending, these results having been obtained on mechanically polished test pieces. Since electropolishing markedly lowered the fatigue limit in rotating bending but had only a slight effect in reversed direct stress, this suggested that the usual discrepancy between Wöhler and Haigh fatigue limits might be due to the greater effect of mechanical polishing on Wöhler test pieces. Accordingly, the fatigue limit in reversed direct stress of electropolished test pieces was determined on two alloy steels, the 13% Cr steel, Code No. HJ1304, and the 20% Cr steel, Code No. 01875. For both, the fatigue limits in rotating bending for electropolished test pieces had already been determined (see Figs. 3b and 4, respectively). The results are given in Table VI.

These results show that the greater part of the difference in fatigue limit between the two methods of fatigue testing is removed by electropolishing both sets of test pieces. The fact that for both alloys the fatigue limit

Fig. 16. — Wohler fatigue fractures of the 3% Ni steel, Code No. 65937, after ½ hr./850° C./B.Q. + 1 hr./650° C./F.G.:
(a) as electropolished; (b) as mechanically polished and stress relieved. All three were tested at ± 30 tons/sq.in.



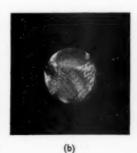




TABLE VI.—FATIGUE LIMITS ON HAIGH AND WOHLER TEST PIECES.

Alloy	Method of Fatigue Testing	Surface Finish	Fatigue Limit (based on 25 × 10 ⁵ cycles) (tons/sq. in.)
13% Cr	Rotating Bending	Mechanically Polished	± 35
13% Cr	Rotating Bending	Electropolished	± 29
13% Cr	Reversed Direct Stress	Electropolished	± 27
20% Cr	Rotating Bending	Mechanically Polished	± 19
20% Cr	Rotating Bending	Electropolished	± 15
20% Cr	Reversed Direct Stress	Electropolished	± 13

in reversed direct stress is still 2 tons/sq. in. lower than that for rotating bending is possibly due to the much larger volume and surface area of material subjected to the maximum stress in the former than in the latter. The area factor would increase the probability of finding a weak spot in the Haigh test piece, at which failure could commence, while the volume factor would ensure a more rapid propagation of a crack.

Discussion

The fact that electropolished Wöhler fatigue test pieces have shown consistently lower fatigue limits, and frequently shorter fatigue lives, than mechanically polished ones for all the alloy steels investigated, can be explained by the following hypothesis: mechanical polishing results in an artificially greater fatigue strength, due to cold work effects which the former introduces to the surface of the fatigue test piece, while electropolishing gives truer fatigue strength values since it reveals a true virgin surface of the material. This hypothesis is supported by the following evidence and arguments:

(1) The consistency with which greater fatigue strength for mechanically than for electrolytically polished Wöhler fatigue test pieces was obtained for the fairly wide range of alloy steels in the present investigation, and also for 1% Cr-V², $2\frac{1}{2}\%$ Ni-Cr⁵, En 25 ($2\frac{1}{2}\%$ Ni-Cr-Mo)⁶, plain 0.35% C⁸, and plain 0.61% C² steels. Since emery polishing clearly involves local plastic deformation, all test pieces so prepared should show some effect of this and, in fact, do so. The near absence of a surface finish effect for the 9/3/8/0.6 Mn-Cr-Ni-C steel at the higher stresses (see Fig. 6) is possibly due to the marked work hardening characteristics of the steel. mechanical polishing has raised the fatigue limit to ± 24 tons/sq. in., which in any cycle is about 2 tons/sq. in. higher than the static 0.5% proof stress of the material. Since stresses higher than 24 tons/sq. in. would involve significant amounts of plastic deformation, the actual local stress conditions during the test may well differ from the nominal applied ones,9 so that the form of the S-N curve may be of doubtful significance.

(2) X-ray diffraction examination clearly confirmed that the surface of an emery polished fatigue test piece was in a cold worked condition, while an electropolished surface was free of these effects. In addition, emery polished surfaces have been shown to contain residual compressive stresses^{2, 3, 4}, which would increase the fatigue strength.

(3) For at least one of the steels investigated, cold work applied to the entire body of the test piece was found significantly to increase the fatigue limit. This increase was observed for electropolished test pieces compared with similarly polished but not cold worked ones. Uniform cold work by stretching has also been shown to increase the fatigue limit of an unstable austenitic 18/8 Cr-Ni steel similar to the one used in the present investigation. This increase was probably due to

the marked strengthening of the steel resulting from the martensitic transformation of the austenite induced by the cold work. The latter explanation would also apply to the similar alloy in the present work. Uncertain results were obtained by the same authors for a stable austenitic 15/28 Cr-Ni steel, possibly due to the smaller strengthening effect of the cold work compared with that obtained in the 18/8 Cr-Ni steel. While no references have been found in the literature for the effect of cold work on the fatigue limit of 13% Cr or 3% Ni steels, increased fatigue limits have been reported for plain carbon steels 10-12.

(4) For the majority of the alloys investigated, and at sufficiently high stresses, the fatigue fractures of electro- and mechanically polished Wöhler test pieces commenced at many, and only one or two, points round the circumference, respectively. This can be explained by a mechanically polished surface being one which is artificially strengthened to resist fatigue. Thus, the probability of finding a weak spot in such a surface is relatively small, while for an electropolished surface it will be large. For fine grained material, the number of grains at the surface in the region of minimum diameter will be large. The number of potentially weak grains will also, therefore, be large. The 9/3/8/0.6 Mn-Cr-Ni-C steel was again apparently anomalous here, in that mechanically polished test pieces also tended to show multi-origin cracking. When surface cold work effects are removed, as by stress relief treatment, the multiorigin failures subsequently obtained are evidence for the opposing influences of cold work and notch effect introduced by mechanical polishing with emery paper. For a fine emery polished surface, the cold work effects are dominant.

(5) Vacuum stress relief treatment applied to mechanically polished test pieces resulted in shorter fatigue lives and lower fatigue limits to an extent similar to that obtained by electropolishing. These results were predicted from the foregoing conclusions about the cold worked state of a mechanically polished surface, and their being obtained helps to confirm the validity of these conclusions. It cannot be claimed that the material in general was weakened by the stress relief treatment, since comparative tests were run on material given the stress relief treatment prior to mechanical polishing. In addition, tensile tests showed no significant change in strength as a result of the stress relief treatment. It is difficult to separate the roles of surface strengthening and surface compressive stresses resulting from mechanical polishing. Both would be expected to increase fatigue strength, and stress relieving as carried out in the present work would tend to reduce both.

(6) Electropolishing had a much smaller effect on the fatigue limit in reversed direct stress (Haigh test) than in rotating bending. This can be explained by the fact that in the latter test it is only the surface which is subject to maximum stress, and its condition is, therefore, more critical than that of a Haigh test piece.

(7) The well known discrepancy in fatigue tests whereby the fatigue limit in rotating bending is generally higher than that obtained in reversed direct stress is largely removed by electropolishing both sets of test pieces. The discrepancy can now be explained as having been due to the greater effect of cold work from mechanical polishing on the Wöhler than on the Haigh type test piece.

(8) The tendency for there to be less scatter of results for electropolished test pieces can be explained by the removal of the personal factor in mechanical polishing, i.e., there is better reproducibility of surface finish in electropolished than in mechanically polished test pieces. This confirms Massonnet's results.8

Conclusions

The present work has shown that mechanical polishing of Wöhler fatigue test pieces artificially raises the fatigue limit and fatigue life due to cold work effects which it introduces to the surface of the test piece. Truer values of fatigue strength are obtained by electropolishing the test piece prior to test. Scatter of fatigue results is also thereby reduced. The well known discrepancy between mechanically polished test pieces tested in rotating bending and reversed direct stress is also largely resolved by electropolishing both sets of test pieces.

Acknowledgments

The author gratefully acknowledges the assistance of his colleagues, and in particular, Mr. A. Goodwin, who carried out the fatigue tests. He also wishes to thank Dr. C. Sykes, F.R.S., Director of Research, Brown-Firth Research Laboratories, for permission to publish this paper.

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Unionmelt Welding of Pipes

COMPLETE Unionmelt welding installation for welding longitudinal seams of pipes has been supplied by Quasi-Arc, Ltd., to Clayton, Son and Co., Ltd., of Leeds. The equipment consists of a Unionmelt fixed internal boom for welding internal longitudinal seams from 18 in. min. i/d. up to 24 ft. 6 in. in length, and a fixed cantilever welding head positioner fitted with a Unionmelt D.S. head, which can carry out external longitudinal and circumferential seam welding of pipes from 12 in. min. o/d. up to 6 ft. max. o/d., and up to 24 ft. 6 in. in length.

The pipes are supported and traversed at controlled welding speeds past the welding head by means of a roller bed comprising a traversing drive and a traversing idler unit. The internal boom welding head has "Raise/ Lower" and "Fore/Aft" fine adjustment, for positioning the wire accurately over the seam. It can also rotate round the horizontal axis of the boom to permit it to follow a longitudinal seam which is not perfectly straight. In addition, a guide wheel is positioned at the rear of the nozzle for accurate guiding along the seam. At the front of the machine, there is a support leg, for

use when it is not in operation.



A platform is attached to the cantilever arm to carry both a melt recovery unit and the operator. Two wire reels mounted on top of the control panel on the cantilever reduce change-over time when the supply of wire on one reel has been exhausted. This arrangement also simplifies the procedure for adapting the welding head for parallel welding, where two wires are fed simultaneously through a single nozzle assembly.

New Craft Training School

A NEW craft training school has been opened for apprentices employed by British Oxygen Engineering, Ltd., at Angel Road, Edmonton. Since 1951, when the Company's new apprenticeship schemes were first put into operation, the need has been felt for a training school completely separate from the production workshops. It will provide apprentices embarking on a five-year course with the firm with a real knowledge and appreciation of basic engineering craft skills, and will enable then to assimilate more quickly the training they receive in the production workshops and technical offices, so that they can carry out their jobs with maximum efficiency. The school will also be used for advanced craft training, and for third year apprentices, to ensure that experience gained in the previous two years in the workshops is firmly established in the minds of the apprentices, that misconceptions can be rectified, and that new developments can be taught.

The school was completed in nine months at a cost of £26,000, the main contractors being Messrs. Kyle, Stewart (Contractors), Ltd., and the architect Mr. F. E. Harvey, A.R.I.B.A., Senior Architect of British Oxygen Engineering, Ltd. The building comprises a machine shop, fitting sections and a lecture room, and the shops are laid out to enable the wide range of work in plant, industrial, medical and aircraft fields to be covered. The school can also be used for intensive courses for the up-grading of fitters, machinists and inspectors, and for the teaching of new techniques to staff in general. The

new building caters for 45-50 students.

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View of Inco's main exploration camp at Moak Lake.

Adequate Nickel Supplies in Sight

International Nickel's Developments in Northern Manitoba

LANS for the development over the next three to four years of a \$175,000,000 nickel project in the Mystery-Moak Lakes area in Northern Manitoba, were announced early in December by Mr. D. Campbell, Premier of Manitoba, and Mr. H. S. Wingate, President of the International Nickel Company of Canada, Ltd. International Nickel will open two new nickel mines in the area, to be known as the Thompson and the Moak Mines, and the project will constitute the biggest nickelproducing operation in the world next to Inco's operations in the Sudbury District of Ontario, and will be the largest single investment of any kind in Manitoba. By arrangement with the Manitoba Government, the new town and the plant site, as well as the immediately adjacent mine, are to be named Thompson in honour of Dr. John F. Thompson, Chairman of the Board of Inco, who this year completed 50 years of service with the company.

The project in Manitoba, together with the progress under way at Sudbury, will lift Inco's regular 1955 annual nickel-producing capacity by approximately 50% to 385,000,000 lb., of which some 24,000,000 lb. will be regular production to replace existing temporary premium-priced production for the United States national stockpile.

Financial Arrangement

The joint announcement was made with the conclusion of understandings between the Province and Inco, including an agreement for the establishment of a new local government district of Mystery Lake and an agreement with the Manitoba Hydro-Electric Board. The latter agreement provides that the Board will undertake construction, at an estimated cost of \$32,000,000–38,000,000, of a large power plant at Grand Rapid on the Nelson River to supply power for Inco's new facilities. The arrangement reflects the long-standing desire of the Government to develop this site, as the capacity of the hydro-electric plant will be sufficient to supply Inco's initial and subsequent power needs, with reserves for other new industries, thus playing its

part in the broad development of Northern Manitoba.

The company will assist the Province in financing the

power plant by way of a four-year \$20,000,000 loan, during the construction period, bearing interest at 2%. Thereafter, the loan will be converted into debentures repayable in instalments over a period of 20 years.

In order to get the programme under way immediately, Inco with its own funds will finance all of the initial capital costs of the undertaking, but expects ultimately to be reimbursed for its advances for the construction of the power plant and for advances which may be made for the new 30-mile C.N.R. spur line. As presently projected. Inco's capital investment in Manitoba, not including expenditures already made for exploration and preproduction items, will approximate \$115,000,000. This will include the cost of opening and developing two mines, of constructing a concentrator, smelter, refinery and transportation facilities, and of providing certain town-site installations. The \$115,000,000 is exclusive of the \$32,000,000-38,000,000 power plant costs, of approximately \$5,000,000 of costs for the new C.N.R. spur line, and of some \$20,000,000 estimated costs of homes and buildings at the town-site.

Mr. Wingate said that in setting up its expansion programme Inco had taken into account the published invitation by the Office of Defence Mobilisation of the United States Government to companies inside and outside the United States for the development of plans for new and expanded nickel production facilities. He said that in response Inco had submitted a proposal to the United States Government covering the expansion projects now announced and the circumstances under which a portion of the nickel from Inco's new and additionally expanded facilities might be supplied, at market prices, to the United States Government.

He stated that while it had not accepted Inco's proposal, Washington had confirmed that if it determines to enter into contracts for new nickel production in Cuba, in connection with its invitation for proposals for expanded production, consideration would also be given to providing Inco with the right to deliver some

portion of its new Canadian production to the United States national stockpile. With this confirmation, Inco is proceeding immediately to initiate these extensive expansion projects, in order to avoid substantial delays by taking advantage of the winter freeze-up for trans-

portation of construction equipment.

Decision to proceed at the present time means a major saving of time in getting the project under way. A month's delay in initiating the project could have postponed the entire programme for a year, through inability to take advantage of this winter's freeze-over in order to haul in over the ice by tractor train much of the necessary heavy equipment required to push the construction forward vigorously in the spring. Pending construction of adequate transportation facilities, this is the only method of moving in such equipment and supplies.

Location of New Orefield

The new Inco mine sites are located about 400 air miles north of Winnipeg and 950 miles north-west of the company's operations in the Sudbury District. They are in an area from 30 to 50 miles north of Thicket Portage, which is on the Canadian National Railways' line between Winnipeg and Churchill, on Hudson Bay. The power plant will be located about 50 miles north-east of the mining operations and approximately 10 miles north-west from the present C.N.R. Hudson Bay line. Moak Mine and the Thompson Mine and town-site will be linked together by a 20-mile company-operated railway and a new 30-mile C.N.R. spur line will connect the Thompson town-site with the C.N.R. Hudson Bay line

from a point north of Thicket Portage.

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The Thompson town-site will be located within the local government district of Mystery Lake, which will be established under Manitoba law and governed by a provincially appointed administrator. Inco will supply school and hospital facilities and other services for the town, which is being planned to accommodate an initial expected population of some 8,000 persons, inclusive of a working force of approximately 2,000. In planning the Thompson town development, Inco will have the benefit of its own past housing and new town building experience, as well as that of municipalities which have been established in other mining areas of Canada in recent years. It is anticipated that an employee-owned home programme will be financed principally through provisions of the National Housing Act, with certain co-operative assistance by Inco.

Initial Production in 1960

The new mining development will contribute greatly to the very much larger supply of nickel which the free world can expect by 1960. The opening up of the Manitoba project in 1960 is part of a major expansion programme by which Inco expects to attain an annual overall regular nickel production capacity of 385,000,000 lb. from its operations in the Sudbury District of Northern Ontario and in Manitoba. In 1955, total Inco Sudbury District nickel production from its own ores was nearly 280,000,000 lb., consisting of approximately 255,000,000 lb., of regular production and 24,000,000 lb. of special production from high-cost ores for the United States Government stockpile.

Since the Manitoba ores carry very small copper values, Inco's copper production capacity will not be increased correspondingly with the large increase for

nickel. Heretofore the copper production has been nearly as large as nickel production. In view of the costs of separating and refining the copper from the Manitoba ores, the copper content is not regarded as a commercial asset. Minor quantities of cobalt, platinum, palladium, gold and silver are present to assist in making the

project successful.

Mr. Wingate forecast that, on the basis of Inco's programme and the announced expansion plans of others, total free world nickel capacity some four years from now may be of the order of 600,000,000-625,000,000 lb., or an increase of about 175,000,000-200,000,000 lb. from all sources above the total of 427,000,000 lb. for The forecast is exclusive of the prospect of increased Cuban output beyond the 50,000,000 lb. annual capacity which the United States Government will shortly have available at its own plant in Cuba. With the achievement of this world capacity, the total free world capacity for nickel supplies should exceed the entire free world civilian consumption of nickel during the 12 months ended September 30th, 1956, by more than 325,000,000 lb. This large amount will be available for the unsatisfied and expanded needs of civilian industry and for whatever the defence requirements may be at that time.

In order to sustain the projected enlarged capacities of Inco and other nickel producers, a considerably increased demand will be needed, and hand in hand with its extensive production project and continuation of its active exploration for still further sources of nickel, Inco is going forward energetically with its work of market development and research, so as to do everything possible



Map of Northen Manitoba showing location of new Inco



Supplies being unloaded from aircraft to tractor-sleigh on lake near exploration camp.

to stimulate the growth of further markets and uses for nickel. In the face of the need for greatly expanded future demand for the metal, the company hopes that other producers will not confine their efforts solely to the production of nickel, but will make a major contribution also by actively pursuing long-range research and development of uses and markets.

Climax to Decade of Exploration

The new mining project climaxes 10 years of exploration in Manitoba, in which particular attention was given to the Mystery-Moak Lakes region, in the belief that this area could be the most likely and strategically situated location for the development of large new nickel supplies. In 1952, the activities there were stepped up further in response to the United States Government request in connection with its consideration of the possibility of assisting in the development of this and other areas. Airborne geophysical exploration, in which as many as 24,000 miles have been flown in a single year, eventually led Inco geologists to the Moak Lake district, after a trapper-prospector's find had brought them to neighbouring Mystery Lake, where subsequent diamond drilling located favourable mineralisation. The total cost which Inco has incurred in its Manitoba explorations is some \$10,000,000.

It is too early to make a definite estimate of the final ore possibilities, but sufficient ore has already been found to justify fully both the money already spent in exploration and the large amounts which will have to be spent to open these properties and put them into production. Current exploration activities have shown that there are fairly wide variations in the grade of ore in different parts of the deposits, but by mining them on a schedule based on the policy of continuous operation over many years, it will be possible to mine an average grade of ore which will compare favourably in nickel content with the ores which have been mined in Sudbury for some years, albeit with much lower copper contents.

The territory over which favourable mineralisation has been found is some 75 to 80 miles in length, with the width averaging approximately 5 miles, and further extensive exploration work will be necessary within the limits of this area. In the interests of the nickel industry and the future of Manitoba, the company's objective is

to develop tonnage and grade potentials sufficient to maintain large nickel production and the longest possible future life of this area, in the same way that these have been possible at Sudbury.

In the past 10 years, prospecting and investigation of this area by others has resulted in only limited interest in its possibilities. Inco's pioneering methods of exploration and its determination, in spite of repeated disappointments, to take major risks and to leave no stone unturned in seeking workable deposits of nickel to add to the world's supply, culminated in making this project possible.

The development will result in no change in the company's extensive exploration programme for nickel, not only in the area outlined by Inco's claims in Manitoba but elsewhere. Thus the company does not rule out the possibility, as progress is made with the present project.

that further expansion may be initiated before the Thompson and Moak Mines come in with the presently scheduled production.

Eighteen diamond drills are in action at present in the Mystery-Moak Lakes area, operating both underground and on the surface. Additional driving is in progress on the 1,300-ft. level in order to explore the orebody at deeper horizons. It is planned to use the exploration shaft as an entry for the development of the orebody. About 235 men are currently employed at the shaft or on diamond drills in the area.

Opening a Frontier

Apart from its importance as a new producing area which will substantially increase the free world's supply of nickel for industry and defence, this development may play an important part in opening up what may well prove to be one of the greatest remaining frontiers in North America. Today some 150,000 square miles in Northern Manitoba surrounding the site of Inco's operations are almost totally devoid of industrial production. The entire area will feel the influence of this great industrial project, and the future changes may be far-reaching in their effect.

British Oxygen Yorkshire Factory

A further stage in the expansion programme of British Oxygen Gases, Ltd., was reached recently, when the Duke of Devonshire officially opened the Company's new factory at Brinsworth, near Rotherham. During the opening ceremony, the Duke set in motion a liquid oxygen producing plant, which is one of six new large plants which the Company is installing in various parts of the country to meet the increased demands for gases.

This new plant is part of a large industrial project at Brinsworth which will ultimately cost £2 million. Building has been in progress for three years, and to date, over £1 million has been spent in erecting an oxygen-producing factory, two plants with storage facilities, and an office block where the Company's Yorkshire District administration is centred. Within two years, the Company plans to build an oxygen compressing station, a dissolved acetylene factory, a loading dock, a garage to accommodate 60 commercial vehicles, and a works demonstration and service block.

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Metallurgy in the Nuclear Power Industry

By W. E. Dennis, B.Sc., D.I.C., Ph.D.

(Chief Metallurgist, The G.E.C./Simon-Carves Atomic Energy Group.)

As in many other fields of engineering, progress in nuclear power production will depend increasingly on the development of suitable constructional materials and methods. The author describes the way in which certain metallurgical aspects are being tackled by his own company and refers briefly to future requirements.

EARLY in 1955 a number of British firms were faced with the task of preparing designs for complete nuclear power stations within a period of eighteen months. For these stations it was required that the source of power should be two graphite-moderated gas-cooled reactors fuelled with natural uranium. This requirement together with the very limited time scale dictated that, at any rate in outline, the design must be similar to that evolved by the U.K.A.E.A. for Calder Hall.

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It was further required, however, that these industrial reactors should be optimised for power production, whereas the Calder Hall plant had been designed for the production of plutonium with power as a by-product. To achieve this, it has been necessary to raise the temperatures of the fuel element, coolant and other materials considerably above those previously employed.

One of the more difficult problems facing the reactor designer is that, owing to the health hazards resulting from irradiation, within a short period after commissioning the plant it becomes impossible to inspect or maintain many of the reactor components. Thus, in designing this plant, which must be capable of satisfactory operation over a period of not less than twenty years, it was essential that data on the probable behaviour of all the materials to be used, under service conditions, should be rapidly obtained.

The aim of the present paper is to describe the way in which this problem was approached by the author's own company, and to illustrate the type of metallurgical work being done in this new industrial field.

The decision was taken in May, 1955, to create a research and development group on materials at Erith, to work alongside the design teams. By August, 1955, the first materials research laboratory had been constructed and a small team of metallurgists and chemists had been recruited. A second laboratory has now been equipped and fifteen graduates are engaged on investigations in these laboratories and on work with the U.K.A.E.A.

In the course of the past year this group has fulfilled three principal functions.

- Carrying out experimental work to test materials under simulated service conditions, and to develop new materials and methods of fabrication where required.
- (2) Maintaining liaison with the U.K.A.E.A. and other research, manufacturing and inspecting organisations.
- (3) Advising the design engineers on all matters relating to the behaviour of materials.

An attempt will now be made briefly to describe the types of problem which have emerged in the course of this work, and to indicate the most likely requirements of the future.

The Fuel Element

For the high level of neutron economy required in a natural uranium reactor, it is considered necessary to have the uranium in the form of cylindrical bars disposed at regular intervals throughout the moderator core. In order to prevent the escape of gaseous and volatile fission products which would activate the whole of the gas circuit, these bars must be sheathed in a suitable canning material. On the grounds of neutron economy and metallurgical properties, a magnesium alloy was selected as the canning material for both Calder Hall and the industrial reactors. In order to facilitate the transfer of heat from the fuel rod to the coolant gas, the magnesium cans are provided with suitable fins. These fuel elements are positioned axially up vertical coolant channels in the graphite moderator core.

In order to obtain the maximum heat output commensurate with satisfactory operation, the following problems required thorough investigation:—

- The provision of an independent fuel element support to prevent bowing due to creep of the uranium.
- (2) The effect of temperature on the corrosion and evaporation of magnesium in the coolant gas.
- (3) The effect of temperature and stress in the particular ranges being considered on the creep of magnesium alloys.
- (4) The effect of the forces exerted by the flow of coolant gas on the fins, and the compressive stresses due to the weight of the fuel element on the lower end cap.
- (5) The effect of end cap design on the quality of the argon-arc sealing weld.
- (6) The compatibility of other materials in the gas circuit with the canning alloy.

It is outside the scope of the present paper to consider these investigations in detail. However, a discussion of some of the work carried out on corrosion and on compatibility may serve to indicate the type of work involved.

Oxidation of the Canning Material

The oxidation of magnesium in carbon dioxide at high temperatures and pressures may involve any or all of the following reactions, depending on the set of conditions used.

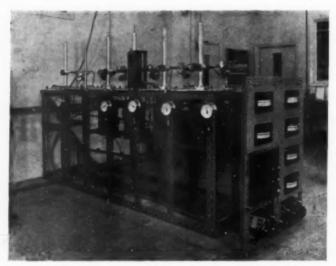


Fig. 1.—A battery of pressurised thermobal inces used for investigating the corrosion of magnesium and its alloys.

From the thermodynamic data it can be shown that under equilibrium conditions with magnesium in the presence of excess carbon dioxide the reaction

$$Mg_{\text{(solid)}} + CO_{\text{2(gae)}} \rightarrow MgO_{\text{(solid)}} + CO_{\text{(gae)}}$$
 (1)

would go to completion.

The reaction,

$$MgO_{(solid)} + CO_{2(gas)} \rightarrow MgCO_{3(solid)}$$
 (2

can be shown to be possible at temperatures up to 420° C. under one atmosphere pressure of carbon dioxide, and up to 525° C. under fourteen atmospheres pressure.

There is also a possibility that under non-equilibrium conditions (e.g., a sudden breakdown of the oxide film), carbon might be produced by the reaction:

$$2 \text{ Mg}_{\text{(vapour)}} + \text{CO}_{\text{2(gaa)}} \rightarrow 2 \text{ MgO}_{\text{(solid)}} + \text{C}_{\text{(solid)}}$$
 (3

Under conditions where the CO/CO₂ ratio exceeds the ratio in equilibrium with graphite, deposition of carbon may also occur, due to catalysis on the magnesium-gas interface.

In the presence of water vapour the reaction

 ${\rm Mg_{(solid)}} + {\rm H_2O_{(gas)}} \rightarrow {\rm MgO_{(solid)}} + {\rm H_{2(gas)}}$ (4) may occur. Under the low concentration of water vapour likely to be present in the coolant gas, magnesium hydroxide cannot form, as it is unstable above 260° C. at less than one atmosphere pressure of steam. The main significance of a small amount of water vapour in the coolant gas lies in its probable role as a catalyst rather than as a reactant.

In addition to these reactions, which would result in a weight increase of a magnesium specimen, the possibility of the process

$$Mg_{(solid)} \rightarrow Mg_{(vapour)}$$
 (5)

occurring at high temperatures has been demonstrated.

Previous workers have studied the oxidation of magnesium alloys in carbon dioxide by measuring the rate at which the specimens gained weight. From the above considerations it is obvious that simple weight measurements cannot be interpreted with confidence in terms of penetration rates. In fact, an observed weight change may be the algebraic sum of gains due to reactions (1), (2), (3) and (4) and losses due to process (5). For this reason, the general picture obtained by the normal procedure is being carefully checked by directly measuring the depth to which the metal has been penetrated, and by determining by chemical analysis the amount of magnesium left and the composition of the oxidation layer.

An apparatus generally known as a quartz spring thermobalance is sometimes used in oxidation studies to allow continuous observation of the weight changes occurring in the specimen during the actual oxidation process. In this equipment, the specimen is suspended on a fine quartz spring and the deflections of the spring are observed by means of a cathetometer, which is sighted through a window situated at the upper end of the furnace tube.

For the present purpose it was necessary to develop an apparatus of this type capable of operating under gas pressures of up to

200 lb./sq. in. A battery of these thermobalances is shown in Fig. 1.

Compatibility

One of the disadvantages of magnesium as a canning material is that it forms low temperature eutectics with the majority of the other commonly used non-ferrous metals and alloys. The use of metals such as copper, aluminium, tin and lead in quantity within the gas circuit is therefore forbidden, because of the danger of contamination causing fuel element failures by eutectic penetration. At an early stage it was realised that under certain circumstances this might prove to be a severe restriction to the reactor designers. A programme of work was initiated to investigate possible methods of protecting magnesium against this form of attack.

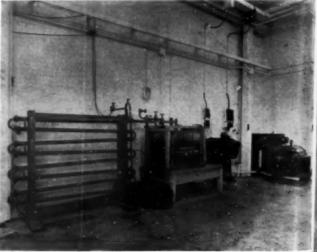


Fig. 2.—Equipment used for anodising and electroplating fullsize fuel elements.

It has been found that by anodising the can in an acid bath of suitable composition, a surface film can be produced which provides protection up to surprisingly high tempera-After some considerable difficulties, tures. a process has now been developed giving a film which is stable under thermal cycling, and which has a low neutron absorption cross During the initial stages of the investigation this work was carried out on small specimens using beakers and batteries. However, in the later stages, in order to obtain data on the effects of these films on neutron absorption cross section and heat transfer, it was necessary to process full scale fuel element cans. The equipment used for this work is shown in Fig. 2.

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The Behaviour of Materials in the Coolant Gas

The coolant gas initially fed into the reactor will be high purity carbon dioxide. During operation it will contain a significant concentration of carbon monoxide, produced by reaction with the graphite core, and some moisture arising from heat exchanger leakages and other

sources. Thus, in assessing the likely behaviour of various materials in the gas circuit, the effect of these impurities has to be considered. In the case of certain problems, e.g.,

- the tendency of nickel carbonyl to be formed by reaction between the carbon monoxide in the coolant gas and components made from nickel and its alloys, and
- the stability of the lubricant molybdenum disulphide,

it was possible to make a preliminary assessment by means of thermodynamic calculations.

In the case of the many different types of steel to be used, it was considered necessary to carry out a thorough



Fig. 3.—Oxidation and other simulated service tests being carried out in one of the laboratories.

programme of oxidation tests in which the effects of temperature, pressure, gas composition and other variables could be evaluated. For this purpose the many hundreds of specimens required were placed in stainless steel pressure vessels, which were maintained at the required temperature by total immersion in standard G.E.C. heat treatment furnaces. A flow of gas of the appropriate composition was continuously fed through these vessels from a system of generators, pipelines and mixing systems. Fig. 3 shows some of this equipment in operation.

In addition to providing the mass of data necessary to enable the designers to make the appropriate allowances for corrosion during service, a considerable fund of information on the characteristics of the corrosion process has





Fig. 4.—Oxidation of Meehanite cast iron in a CO/CO₂ atmosphere at 525° C. Left—after 3,000 hr.; right—after 3,800 hr.



Fig. 5.—Investigators observing the automatic argon-arc welding of a fuel element end cap.

been obtained. In particular, it has been found that the rate of oxidation of some ferrous materials is markedly affected by the presence of a small concentration of carbon monoxide. For instance, in the case of certain types of cast iron the presence of carbon monoxide causes catastrophic corrosion (see Fig. 4).

Fabrication

An important part of the work carried out under this heading has been to ensure, so far as is possible, that all the welding and other joining processes used will give results of the high standard required. Programmes of welding tests on the reactor shell plates have been carried out to optimise on the choice of electrodes and the welding procedure.

Automatic and manually operated welding equipment has been installed in the laboratories (see Fig. 5) and, the effect of design on the quality of welds obtained in the argon-arc welding of fuel element end caps has been studied in detail. The optimum conditions for welding special components in relatively inaccessible positions have been determined by a series of tests on specially constructed rigs.

Future Requirements

Future requirements on materials are expected to fall within two distinct categories. First, problems arising in connection with the present reactor design; and second, research and development work required for more advanced reactors to be designed in the future.

Considerable effort has already been devoted to meeting the requirements which will arise during construction. Ultrasonic and other inspection equipment required on site has been assessed. Experiments have been carried out to determine the best methods of preparing the various reactor components in a state of nuclear cleanliness, and of protecting them from corrosion or contamination during transport and erection, and detailed proposals have been formulated for the chemical and handling plant required for this work.

With regard to future reactors, a survey has been made of the problems associated with the use of hydrogen and sodium as coolants. Most of the equipment used in connection with the present carbon dioxide-cooled reactor design will be readily adapted to work with hydrogen, and plans are being prepared for the construction and operation of a small sodium loop in the near future.

Metallurgical Oxygen Processes

GIBBONS BROTHERS, LTD., a subsidiary of Gibbons (Dudley), Ltd., announces that it has acquired an interest in Metallurgical Oxygen Processes, Ltd., of Granite House, Cannon Street, London, which has developed processes for the heating of metals without scale or oxidation. The present directors, Dr. A. G. E. ROBIETTE and Mr. J. MILES, remain on the Board.

The patented processes controlled by Metallurgical Oxygen Processes involve the use of oxygen, and are designed to effect considerable economies in the iron and steel industry, especially in the rolling, forging and extrusion of steel. Licences have already been granted in the U.S., Canada, France, Belgium, Austria and other countries.

Wickman Wimet Division Expansion

A new office block has recently been completed and occupied by the Wickman Wimet Division at their main works in Torrington Avenue, Coventry. Of reinforced concrete construction, the new building provides about 10,000 sq. ft. of administrative office space on two floors, and accommodates staff displaced from the main building through expansion in manufacturing capacity, and additional personnel to facilitate the handling of an increasing volume of daily orders. The new building represents the completion of the first phase in the Company's new plans for expanding Wimet tool production. Extensions to the Coventry plant constitute the second phase, now in the course of installation, and considerable extension to the Glasgow Works, to be started shortly, will complete the present plan.

British Oxygen Wimpey Ltd.

The British Oxygen Co., Ltd., and George Wimpey, Ltd., have formed a jointly-owned subsidiary to be known as British Oxygen Wimpey, Ltd. The purpose of the new company is the construction of special rocket projects, and the wide experience of the two sponsors in major building contracts on the one hand and low temperature techniques for tonnage liquid gas on the other will enable all phases of a project to be dealt with within the new organisation.

B.S.A. Research Move

THE B.S.A. Group Research Centre has been transferred from Greystones Hall, Sheffield to Mackadown Lane. Kitts Green, Birmingham, 33 (Tel.: Stechford 3657-8-9)

The Repeated Strain Ageing of Mild Steel*

By B. B. Hundy, † B.Sc., Ph.D., A.I.M., and T. D. Boxall ‡

Strain ageing of steel takes place after each individual cold working operation to which it is subjected, and the authors' results show that the effects of all ageing and cold working operations on the mechanical properties are additive. If only the last of several cold working operations is followed by ageing, the effect is less than if the steel is aged after each pass. An explanation is offered, based on the dislocation theory of strain ageing.

NUMBER of cases arise in practice where mild steel is cold worked, allowed to age either at room temperature or above, and then again cold worked and aged. For example, mild steel sheet is usually temper rolled (i.e., rolled to a reduction of 1-3%) before despatching to the press shops, to prevent the formation of stretcher strain during pressing. The steel is not usually pressed for a week or two after temper rolling, and so it becomes partially aged. When this occurs, it usually means that the sheet has to be roller levelled at the press shop before pressing, and this gives another opportunity for ageing to take place. Finally, the sheet is pressed into a component which can then strain age again during service. Fig. 1 shows the effect of ageing on the ductility of mild steel sheet temper rolled 1.7%, aged for one month, and then roller levelled and aged again. It can be seen that the ageing process seems to start afresh after each cold working operation.

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Another example occurs in the drawing of patented high-carbon steel wire for rope. If the reduction per pass during drawing is moderately high and the wire is not cooled between passes, strain ageing due to the heat generated during the working can occur between each pass, and can affect the properties of the finished wire. Table I compares the mechanical properties of such wire drawn to a total reduction of 85% with a reduction per pass of approximately 24%: (a) after drawing with no cooling; (b) after drawing with ample water cooling to reduce the ageing; and (c) after drawing with water cooling and then finally ageing for 48 hours at 58° C. The number of bends and twists to fracture are probably the most sensitive criteria of wire quality, and it can be seen that these are markedly affected by the conditions of drawing. Drawing without cooling (i.e., where the

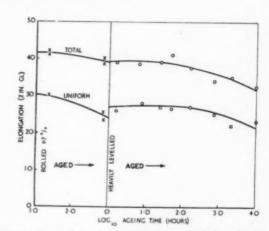


Fig. 1.—The effect of a sequence of working and ageing operations on the ductility of mild steel strip.

heat developed in the wire can cause strain ageing between passes) gives wire with the least ductile properties under these particular conditions.

To the best of the authors' knowledge, very little previous work on the effect of repeated strain ageing has been done. A paper by Edwards, Jones and Walters1 includes some work of this sort, and the figures presented in Table II are taken from this source (in some cases interpolation of results was necessary): it seems from these that ageing after each 2-3% tensile strain has a greater effect on the mechanical properties of the steel than pulling to the total strain and then ageing fully.

Palm² studied the effect of repeated roller levelling (up to 40 times through the leveller) on the properties of a 0.1% C steel, and found that this had no effect. When, however, the strip was aged at 100° C. for six hours after each pass through the leveller, it was found that the strength increased steadily and the ductility decreased

Report No. MW/E/47/56 of the Sheet Metal Working Committee of B.I.S.R.A. † † Steel, Peech and Tozer Branch of the United Steel Companies, Ltd. † British Iron and Steel Research Association, Sheffield Laboratories.

TABLE 1.—THE EFFECT OF AGEING DURING THE COLD DRAWING ON THE MECHANICAL PROPERTIES OF A HARD DRAWN PATENTED 0-7% C. STEEL WIRE. (DRAWN TO A TOTAL REDUCTION OF 85% IN 24% PASSES).

Treatment	Number of Bends R/r = 10	$\begin{array}{c} \text{Number} \\ \text{of} \\ \text{Torsions} \\ 1/d \\ = 100 \end{array}$	Ultimate Tensile Strength tons/ sq. in.	Proof Stress tons/ sq. in.	Reduc- tion of Area at Frac- ture %	gation	Blastic Modu- lus × 10° lb./ sq. in.
No Cooling during Drawing	48	46	111	93	54	0.4	28
Water Cooled dur- ing Drawing	59	51	110	96	36	0.6	28
Water Cooled dur- ing Drawing and then Aged for 48 hr. at 58° C	56	50	119	97	54	0-4	28

TABLE II.—THE EFFECT OF DIFFERENT AGEING TREATMENTS ON THE MECHANICAL PROPERTIES OF COLD WORKED MILD STEEL. (AFTER EDWARDS, JONES AND WALTERS (I)).

	General I	longation	Tensile Strength		
Strain Sequence	Aged After Each Strain (%)	Only Aged After Final Strain (%)	Aged After Each Strain (tons/sq. in.)	Only Aged After Final Strain (tons/sq. in.)	
1.8%	17.0	17-0	24.0	24.0	
1.8% + 2.4%	11-6	13-5	26-6	25-4	
1.8% + 2.4% + 1.6%	3-5	11.5	27-1	26-5	
1·8% + 2·4% + 1·6% + 2·7% + 2·5%	3.0	8-2	32.9	29-6	

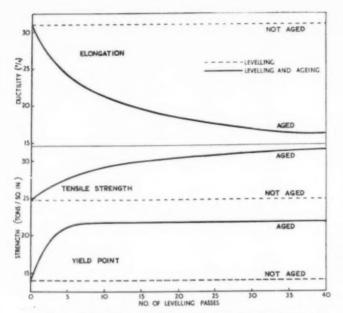


Fig. 2.—The effect of roller levelling and roller levelling with ageing on the mechanical properties of mild steel (after Palm²).

(Fig. 2). This seems to confirm Edwards, Jones and Walters' finding that repeated strain ageing can have a profound influence on the mechanical properties of steel.

Experimental Work

The experiments described here were designed to investigate in a more detailed manner the effect of repeated strain ageing on the mechanical properties of mild steel. It was decided to use mild steel wire and

strain it by drawing, in preference to other methods of straining such as rolling or stretching. This had the advantage of being able to control very accurately the reduction during each pass and to enable comparatively small reductions to be given with ease. This method also had the advantage of simplicity in preparation and testing of the tensile specimens.

The steel used for these experiments was of a low carbon rimming quality (C-0.052%, Mn-0·43%, S-0·034%, P-0·019%). The samples were prepared by cold drawing straight lengths of wire to 12 s.w.g. and annealing subcritically in an inert atmosphere followed by furnace cooling. The samples were drawn through dies having die angles of 12-16° but, as the reductions used were very small (1-2.2%), the effective die angle would be appreciably lower: because of this, the variation of strain through the thickness the of wire would be very small.3 Ageing of the specimens was carried out in an oil bath and the equivalent times of ageing at room temperature calculated,4 1 hour at 135° C. being considered sufficient to give complete ageing. Wires approximately 10 in. long were tested in tension, using an Amsler

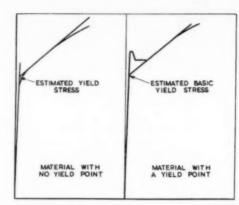


Fig. 3.—Method of extrapolation to obtain yield stress.

Universal testing machine; the initial part of the stress strain curve being recorded autographically. The uniform and total elongation (2 in. gauge length) and maximum stress were measured on all specimens, and the basic yield stress of the wires estimated by extrapolation, as shown in Fig. 3.

Results

It was decided first to examine the changes in the mechanical properties of wire lightly drawn,

aged and then drawn again and aged. Samples of the wire were drawn to a reduction of $1\cdot 2\%$ and specimens were tested after ageing for various times up to the equivalent of two years (Fig. 4). At the end of this period, the rate of change of most of the mechanical properties seems to have slowed down, and it seems unlikely that further change would occur with any lengthier ageing. Some of these fully aged samples were then redrawn $2\cdot 0\%$ and the change in properties on subsequent ageing again observed: Fig. 4 shows the results of these tests also.

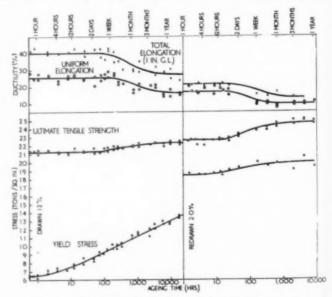
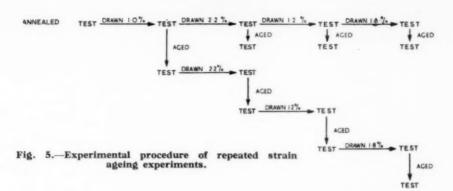


Fig. 4.—The effect of ageing on the mechanical properties of mild steel wire drawn 1·2%, aged, redrawn 2·0% and aged again.



It will be seen that the strain due to the second drawing process has comparatively little effect on the tensile strength or ductility. The ductility and tensile strength of the wires immediately after this drawing are almost the same as those of the fully aged single drawn wires. However, as the ageing is allowed to proceed there is a further drop in the ductility and increase in tensile strength. From this, it seems that although the change in properties due to strain ageing may be almost complete after two years, further plastic deformation enables the steel to age to a greater extent, leading to further changes in the mechanical properties. Samples of wire drawn directly to the total reduction of $3\cdot 2\%$ and then fully aged had mechanical properties as follows (figures in brackets are those obtained with the intermediate ageing treatment after the first reduction of 1.2%):-

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Ultimate Tensile Strength ... 24 tons/sq. in. (25)
Uniform Elongation 14% (10)
Total Elongation 16% (12)

These results suggest that a greater total change in the properties due to ageing can occur if the total strain is given to the specimen in increments, with an ageing treatment after each increment. To study this point more fully, samples of wire were drawn to a total reduction of $6\cdot2\%$ using separate passes of $1\cdot0$, $2\cdot2$, $1\cdot2$ and $1\cdot8\%$ reduction. One series of wires was drawn and tested with an ageing treatment after each pass, whilst another series was tested and drawn without any ageing at all. A third series was tested after drawing with ageing only after the final pass. All ageing treatments were such as to give complete ageing, and specimens were tested at all stages in the processes, i.e., before and after each drawing pass, after ageing, etc. Six specimens were tested in the various desired conditions, and the plan of experiments is schematically shown in Fig. 5. Fig. 6 and Table III show the results of these tests, the figures quoted being the arithmetic average of the six results.

Fig. 6 shows that cold working by drawing alone causes only a moderately steady decrease in the ductility of the wire and increase in the yield strength and ultimate tensile strength. Each ageing treatment causes a further fall in the ductility and rise in the strength properties, and it can be seen that in general ageing gives a greater change in the ductility and tensile strength than does the small amount of cold working. One slight anomaly in these results is the small increase, as opposed to the usual decrease, in the ductility after the final ageing treatment after drawing to 6.2% reduction.

If these results are compared with those given in Table III for wires aged only after the final pass, i.e.,

after $3\cdot 2$, $4\cdot 4$ and $6\cdot 2\%$ reduction, it will be seen that ageing after each pass gives a greater change in the mechanical properties than if ageing is allowed to take place after the final pass only.

It is also to be noted from Figs. 4 and 6 that the increase in the ultimate tensile strength on ageing after each pass is roughly proportional to the reduction of the previous pass, i.e., a large reduction leads to a large increase in the ultimate tensile strength on ageing. The yield stress and ductility figures have a tendency to show the same effect, but in the case of the uniform elongation this is masked by the greater tendency of the ageing effect to decrease as the overall reduction is increased.

To summarise, then, it seems that strain ageing takes place after each individual cold working operation to an

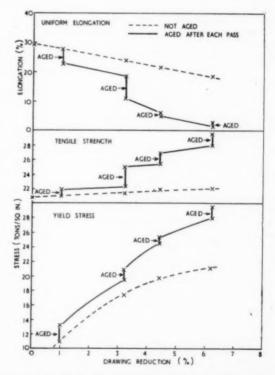


Fig. 6.—The effect of repeated strain ageing on the mechanical properties of mild steel wire. Ageing treatment: 1 hr. at 135° C. (each point shown is a mean of six experimental results).

TABLE III.—THE PROPERTIES OF MILD STEEL WIRE AFTER VARIOUS REDUCTIONS AND AGEING TREATMENTS.

Drawing Reduction		after Each	Ageing after Final Pass Only		
	Elongation (%)	Ultimate Tensile Strength (tons/sq. in.)	Elongation (%)	Ultimate Tensile Strength (tons/sq. in.)	
3-2% (1% + 2-2%)	10	25	14	24	
4·4% (1% + 2·2% + 1·2%)	8	26.9	11	25-3	
6-2% (1% + 2-2% +	0.5	90.5		98.5	

extent dependent on the severity of the reduction preceding ageing, and that the effects of all ageing and cold working operations on the mechanical properties are additive. If the steel is given several cold working passes and aged only after the final pass, then the total change in the mechanical properties is less than if the steel had been aged after each pass.

Discussion

The explanation of the phenomenon reported here can be found in the dislocation theory of strain ageing.⁵ In the annealed mild steel the dislocations are locked in place by "atmospheres" of carbon and nitrogen atoms, and a much larger force is needed to start these dislocations moving (and hence start plastic deformation) than is needed to keep them moving once they are away from the influence of the solute atom atmospheres. This is the explanation of the discontinuous yield point in mild When the steel is deformed plastically, all the dislocations are freed from their atmospheres and the number of dislocations is increased many fold (probably from 107 dislocation lines/sq. cm. in annealed metal to 1010 dislocation lines/sq. cm. in steel cold worked 2-3%). When the steel is allowed to age after this straining, the nitrogen atoms diffuse through the iron lattice and lock all the dislocations (both the original and the new ones) in place and the yield point returns. When each dislocation has attracted more than 1-2 solute atoms per atom plane, it is thought6 that solute atoms in excess of this number diffuse along the dislocation line until they meet some obstruction (probably the junction with another dislocation line), where they form a tiny precipitate. These small precipitates have a hardening effect on the steel and cause the yield point to rise further, the tensile strength to increase, and the ductility to decrease. The effectiveness of these precipitates in hardening the steel is dependent on their size (i.e., on the number of solute atoms attracted to each dislocation, and hence time of ageing) and on their spacing. The spacing of the precipitates will be dependent on the dislocation density: this in turn is proportional to the amount of plastic deformation. This explains the increasing amount of strain ageing with increasing plastic strain (reduction/pass).

On this basis, therefore, one might expect that the amount of strain-age hardening should be independent of whether or not intermediate ageing treatments are given after each cold working operation. At first sight, one might expect the same amount of total strain to give the same dislocation density, and hence the same amount of strain-age hardening. The explanation, however, is that when the mild steel is strained after strain-age hardening, the tiny precipitates formed during the ageing increase the rate of work hardening of the steel, which means that the number of dislocations introduced into

the steel by further straining is greater than if the steel had not been aged before cold working. Thus, a specimen that had been aged several times at intermediate values of cold reduction would have a higher dislocation density, and hence would show greater total strain-age hardening, than a specimen cold reduced to the same amount and only allowed to age after the final pass.

The increased rate of work hardening caused by ageing has been observed previously, when the effect of strain ageing on the shape of the true stress-true strain curve was examined.6 It can also be seen when the rise in yield stress per pass (and to a lesser extent the tensile strength) of mild steel aged between each pass is compared with that of mild steel drawn without ageing (Fig. 6).

Conclusions

If the maximum strain-age hardening effect is desired, i.e., to strengthen the material appreciably for applications where ductility is not important, then the cold working operation should be interrupted as often as possible with intermediate ageing operations. (The cold working operations could be carried out when the steel was warm, 100-200° C.). If, on the other hand, as is usual, the strain-age hardening process is to be minimised as much as possible (e.g., to prevent a very large loss in ductility) care should be taken to ensure that the steel is kept as cool as possible during working, and also not to allow it to age appreciably in between the cold working operations.

Acknowledgments

The authors wish to express their appreciation to Mr. D. Burton and Mr. B. Coldwell for their help in the experimental work.

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Disposal of Non-Ferrous Metal Stocks

In accordance with the policy announced in the Defence White Paper Cmd. 9691 (paragraph 125), of running down the strategic holdings of industrial raw materials. the Board of Trade are planning to reduce their stocks of lead, zinc and tin. The Board are anxious to make these disposals without unduly disturbing markets. In the case of lead and zinc, no sale will be made before the middle of January, 1957, and in accordance with the period of notice stipulated in Article XIVe of the International Tin Agreement, disposal of up to 2,500 tons of tin will not begin before June.

Dollar Order for English Electric

Two of the largest three-phase transformers ever built have been ordered by the Tennessee Valley Authority from The English Electric Company for the Authority's new power station at Gallatin, Tennessee. Their capacity is 345,000 kVA. each, and they are to transform the generator pressure of 22.5 kV up to the transmission voltage of 165 kV. The value of the contract is over £300,000.

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Magnesium and its Industrial Applications

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United Kingdom Facilities for Production and Development



Virgin metal production plant, showing three lines of cells with the two chlorinators in the background.

ECHNOLOGICALLY speaking, magnesium is a comparatively new metal, for although it was first isolated by Sir Humphrey Davy almost 150 years ago, it has only been used on an industrial scale since 1923. Due to high costs of production and difficulties in application, progress was slow, but by 1938 estimated world production had risen to 32,000 tons. Due to inflated wartime demands, the figure rose to a peak of 228,000 tons in 1943-44, but fell to 10,000 tons in 1946, when demand was met mainly from stock. As new uses were developed and stocks absorbed, production slowly increased to an estimated figure for 1955 of 124,000 tons (including U.S.S.R.). Over 80% of this output is accounted for by production in the U.S.A. and the U.S.S.R. Figures for U.K. production during the same period were: 1938-3,000 tons; 1943-18,800 tons; 1946-600 tons; 1955-1,500 tons. In contrast to the figures for production of primary metal, U.K. sales of magnesium and magnesium alloys amounted to 7,500 tons in 1954 and 11,000 tons in 1955.

Historical

The United Kingdom, in 1864, was the first country in Europe to produce magnesium in small quantity for photographic purposes, but the first step to the industrial application of the metal was taken in 1886, when Germany began to produce magnesium commercially on an industrial scale, using carnallite (MgCl₂, KCl, 6H₂O) from the Stassfurt deposits as raw material. It was not until 1909 that the first alloys of magnesium, containing aluminium and zinc, were produced, and although considerable tonnages were used by Germany for munitions in the first world war, their high cost and low corrosion resistance prevented any real progress in this field after the war.

The period 1923-26 was, perhaps, one of the most important in the history of the magnesium industry, marked as it was by three important developments. In 1923, I.G. Farbenindustrie workers at Bitterfeld discovered that magnesium could be melted and refined by using high-viscosity fluxes which absorbed non-metallic impurities such as oxides, nitrides and chlorides, but

which remained sufficiently viscous to avoid retention in the metal. This development was a notable advance in melting technique. In 1925, Boyer, in the U.S., and Beck, in Germany, working independently, made the important discovery that the addition of an excess of manganese to molten magnesium not only precipitated impurities such as iron, but also greatly increased the corrosion resistance of the resulting alloy. The third development concerned the extraction of the metal, and was again due to workers in the Bitterfeld group. Pistor. Moschel and their collaborators, established a process for the chlorination of calcined and raw magnesite with carbon and chlorine, to give a completely anhydrous molten product. The molten magnesium chloride was then electrolysed to molten magnesium and chlorine, which was returned to the chlorination plant. This process, or modifications of it, together with the Dow process developed in the United States, to-day account for most of the world's magnesium production, although a certain amount is produced by thermal reduction directly from dolomite, using ferro-silicon. The main difference between the I.G. and Dow processes is that the latter uses partially hydrated magnesium chloride as cell feed, whereas the I.G. process uses the completely anhydrous salt. In spite of the availability of a number of magnesium-bearing minerals, of which dolomite is the most abundant, by far the largest tonnage of the metal is to-day extracted from sea water (which contains 0.13%-54 m. tons per cubic mile) by the Dow Chemical Co., at Freeport, Texas, and by the Norsk Hydro-Elektrisk Kvoelstofaktieselskab at Heroya, in Norway. Because of inherent natural advantages, these are the two lowest cost producers in the world.

U.K. Progress

In 1862–63, Edward Sonstadt, an English chemist, patented a process for reducing magnesium chloride by sodium and distilling the crude magnesium in hydrogen. The Magnesium Metal Co., Ltd., was formed by Sonstadt in 1864 to make magnesium metal and wire for photographic purposes, but the works closed in 1890 because the product could not compete with German imports at



General view of the production foundry where virgin metal is refined and alloyed before casting into ingots.

considerably lower prices. Apart from production by this process during World War I, there was no further production of magnesium metal in the U.K. until 1936, although various processes were tried unsuccessfully in the intervening period.

During this period a demand for the metal for alloying with aluminium and for industrial and service use was slowly built up by F. A. Hughes and Co., Ltd., and British Maxium, Ltd. (later Magnesium Castings and Products, Ltd.) based on imports from Germany and the U.S.A. respectively. In 1935, F. A. Hughes acquired for the British Commonwealth the patent rights of the I.G. thermal electrolytic extraction process, and formed Magnesium-Elektron, Ltd., as the operating company, with I.G. and I.C.I. as the other shareholders. In 1936 a works was erected at Clifton Junction, Manchester. which was designed to extract 1,500 tons of metal per annum from imported raw and calcined magnesite. This plant was put into operation in December, 1936, and a further 2,500 tons per annum capacity (at Government cost) began production in January, 1938. In 1941, a further 5,000 tons per annum unit was commissioned and in 1942 the Government instructed M.E.L. to erect a 10,000 tons per annum plant near Burnley, which started up in 1943.

The first electrolytic plant in the U.K. used raw and calcined magnesite imported from Greece, Yugoslavia and India, but from 1938 onwards, magnesium recovered from dolomite and sea water became increasingly available. The British Periclase Co. was the first in the U.K. to treat sea water with calcined dolomite to recover the magnesium in both. The use by M.E.L., in March 1939, of magnesia from the British Periclase Co., was the first recorded commercial production from sea water. The magnesia obtained by this process was of a highly reactive causaic type, well suited for chlorination to anhydrous magnesium chloride, and 40,000 tons per annum were produced during the war years.

During the decade, 1935–45, a number of thermal reduction processes were operated, with varying amounts of success, using such agents as calcium carbide, carbon and ferro-silicon. After the war, these plants were

closed down and the electrolytic plant put on "care and maintenance," until, by 1947, there was no longer any production in the country. By 1951 the stock of pure magnesium had been used and the Government instructed M.E.L. to re-activate a 4,000 ton electrolytic unit, which continued in operation until early 1953, when Government support ceased. Part of the plant was again shut down, but M.E.L. found a way to continue producing on its own account at the rate of 1,500 tons per annum in its original plant.

The high cost of electric power, which today is five times the 1938 figure, makes it impossible, without subsidy, to operate the established electrolytic processes and compete with magnesium available from the United States, Norway and Canada. Intensive efforts are being made at M.E.L. to develop a method of extracting magnesium from indigenous dolomite deposits with the use of U.K. power, and considerable progress has been made.

Production at Clifton Junction

To-day, Magnesium Elektron, Ltd., is the sole producer of virgin magnesium in the United Kingdom. It is also virtually a self-contained unit for the fabrication, development and servicing of magnesium from the ore to the finished product. Indeed M.E.L's knowledge of the metal in all its aspects is unrivalled in Europe and acknowledged throughout the world. The following brief description of the facilities at the Clifton Junction works of the company will serve to indicate the scope of its activities.

Chlorination and Electrolysis.

As mentioned earlier, magnesium is produced by the electrolysis of magnesium chloride, which is obtained by the chlorination of magnesia. Both these operations are carried out in one building, which houses two chlorinators and three lines of electrolytic cells. The chlorinators comprise refractory-lined cylindrical chambers, charged at the top with pellets composed principally of magnesium oxide and carbon. The bottom section of the reaction chamber is filled with carbon resistor block packing and power is fed in by means of six electrodes. Chlorine is fed into this section and rises up the stack, reacting with the pellets to produce magnesium chloride and carbon monoxide. Reaction occurs at temperatures approaching 1,200° C., and the molten magnesium chloride trickles down through carbon resistor block packing, collecting as a molten pool in the bottom, whence it is tapped into tilting crucibles for charging. still molten, into the cells. Originally, all the chloride required was produced in this way, but to-day, owing to increased costs, some of the chloride is brought in and charged directly to the cells.

The electrolytic cells are connected in series, the voltage drop across each being approximately 7 v. The D.C. current used is 18,000–20,000 A., and each cell produces over 400 lb. of magnesium each day. The metal is deposited at the cathodes in molten form and being light, floats on the surface of the cell melt and is ladled out once every 24 hours. The chlorine is con-

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81 di inuously exhausted from the cell, some being used in the chlorinators for the production of more chloride, the rest being burnt in producer gas and water vapour to produce eventually hydrochloric acid solution, some of which is used internally for the production of zirconium compounds, and the rest sold as such.

Ingot and Billet Production.

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Melting and refining of the virgin metal is carried out in the production foundry, where magnesium alloys are also made and cast into ingots; scrap metal is similarly treated. Melting equipment includes two 2-ton and a number of 300-lb. crucible furnaces fired by producer gas, and melting is carried out under a protective flux. The metal is poured from a crucible held in a lødle carriage of the lipaxis tilting type, onto a continuous belt of ingot moulds, the stream of metal being subjected throughout the operation to a sulphur dusting to inhibit oxidation.

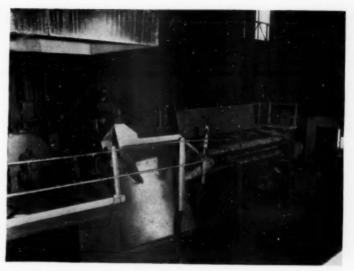
Billets and anodes of various shapes and sizes are also cast in the same shop. In billet casting the moulds lie almost horizontal, and pouring is slow to avoid oxide inclusions. As pouring nears completion, so the mould inclines to the vertical. The billets, etc., are fed with hot liquid metal to compensate for shrinkage. So-called "book" moulds are used for the casting of slabs for subsequent rolling down to sheet.

For casting billets and slabs up to 800 lb. in weight and 102 in. in length, there is a direct chill casting machine of the semi-continuous type. The liquid metal is poured into a shallow mould whose base is a descending platform. As the metal emerges from the base of the mould it is directly chilled externally by impinging jets of water, causing the shell to solidify immediately, although the core remains initially molten. The rate at which the platform descends can be varied according to the alloy cast.

Billets and slabs for wrought products have to be machined. This is carried out on one or other of the machines in the foundry machine shop, and the phenomenal operating speed is noteworthy, being twice as fast as for aluminium and ten times the speed for steel. A special Swift lathe with a spindle speed of 3,000 r.p.m. removes the outer metal surface. It traverses at 27 in./min., taking a cut ¼ in. deep. Other equipment consists of two circular saws, a large band saw, and a special milling machine for skimming, two at a time, the major faces of rolling slabs.

Flux Production.

Another section of the manufacturing plant is concerned with the production of fluxes for use in melting, refining and alloying. Zirconium, for example, is introduced into molten magnesium by means of zirconium-containing salts. Again, since loss of both zirconium and rare-earth metals occurs on melting, processes have been devised for the revivification in a simple manner of both the zirconium content and the rare-earth metal content by the addition to the melt of special salts, so that foundry personnel have little more difficulty in handling zirconium alloys than those of the older types.



Ingot casting machine in the production foundry. Billets and anodes of various shapes are also cast in this foundry.

Research Facilities

The company prides itself on being technically progressive, and past results have indeed proved the wisdom of wholeheartedly supporting extensive research and development programmes. Owing to the very wide technical field covered, the company's research is carried out in two departments, one specialising in chemical and the other in metallurgical problems.

Chemical.

Because the high cost of electricity and materials has rendered the electrolytic method uneconomic, one of the more important tasks of the Chemical Research Department is the development of other methods of extracting magnesium. One of the possibilities in which the company has high hopes is the reduction at high temperature and under vacuum of briquettes made of calcined dolomite (which occurs extensively in the U.K.) and ferrosilicon. The magnesium metal is produced as vapour and is condensed on the cool ends of the retorts. This process is at present being studied on a pilot plant scale.

Metallurgical.

The Metallurgical Research Department is responsible for the development of new alloys of improved mechanical properties, for the investigation of high purity alloys, and for work on powder metallurgy. Its functions include X-ray and spectrographic work for the control of sand foundry products and of billet and slab casting quality. A separate corrosion laboratory is maintained, and extensive work on surface protection processes is in progress.

For the routine testing of the efficiency of the batches of master salts, there are six small melting furnaces in an experimental foundry. These furnaces are also used for the production of experimental alloys, which can subsequently be extruded on a 400-ton press, or rolled in a small mill. Both these units are primarily used for experimental purposes, but they also find application for small scale production.

The mechanical testing laboratory contains equipment



Direct chili machine for the semi-continuous casting of billets and slabs up to 800 lb, in weight and 102 in, in length.

for the tensile testing of specimens at room and elevated temperature, for shear testing, for hardness testing, and for fatigue testing of rotating cantilever specimens. There is also a machine for fatigue testing at elevated temperatures.

Equipment of various types for measuring resistance to creep deformation at elevated temperature is housed in a specially built laboratory, which also contains a machine for fatigue testing, using either plain tensile or a combination of tensile and compressive stresses. Because of the necessity of maintaining accurate temperatures, this room is thermostatically controlled.

All stressed aircraft castings are required to be examined radiographically, and for this purpose a 140 kV. industrial X-ray set is available. The equipment

of the X-ray section also includes a crystallographic unit for use in investigating the structure of alloys. Apart from a normal chemical laboratory, there is a spectrographic analysis section equipped with three spectrographs of the conventional type, in which the intensity of the various lines is assessed photographically, and a Quantometer. The latter is a direct-reading spectrograph which gives a complete analysis of a magnesium alloy in about a minute, the analysis of an alloy being thus available before the metal is poured into ingots. Essentially, the principle involves direct measurement of a very small amount of light emitted at selected wavelengths from each of the elements present in the alloy, when subjected to the condition of an electric arc or spark, the results being recorded on a chart. The atmosphere in the spectrographic laboratory is controlled from the point of view of both temperature and humidity.

Prototype Fabrication and Founding

Not unnaturally in a company concerned with a metal whose use is expanding, con-

siderable attention is paid to new applications and new methods of fabrication, particularly of the wrought material. Evidence of the multifarious activities carried on in the fabrication shop is provided by the equipment assembled therein: lathes, drills, milling machines, presses, bending rollers, welding sets, a painting booth, and a stoving oven. These fabrication facilities are being greatly expanded at present, and the changes will greatly influence prototype and production work, and the manufacture of fabricated assemblies, pressings, spinnings, and all types of magnesium sheet work. A 380-ton hydraulic press is being installed, together with other sheet-working equipment, and in the near future this section is to be expanded to a Fabricating Division. There is to-day a growing demand for wrought magnesium in airframes, in welded oil tanks, in guided missiles, and in radar and electronic equipment and this will be met by the facilities to be provided.

The sand foundry, too, is devoted mainly to experimental and prototype work, although production work is also undertaken,

the main lines being for textile machinery accessories. Most of the prototype work is of a "classified" nature—radar and guided missile parts, engine castings, etc. It was in this foundry that the castings for the upper atmosphere research rocket, to be launched by Britain in connection with the International Geophysical Year, were moulded and poured in ZREI alloy. Development work carried out in the foundry includes studies of casting properties and investigations into foundry methods suitable for specific alloys. The equipment includes eight melting furnaces, sand preparation machinery, core-making facilities, core-drying ovens, sand-blasting equipment, and fettling and grinding tools (including dust exhaust).



Batteries of machines for testing the resistance of magnesium alloys to creep deformation are housed in a specially built laboratory.

Alloy Development

One of the principal attractions of magnesium is its ultra-light weight-approximately one-quarter of that of iron and steel and two-thirds that of aluminium-combined with relatively high strength. Although the elastic modulus of magnesium is lower than that of other metals, an increased rigidity and simplification of structure, with considerable saving in weight, can be obtained by thickening up the sections in magnesium, thus avoiding the need for additional stiffening elements, with their attendant assembly labour costs. Another attractive feature of magnesium and its allovs lies in their machinability, which enables them to be machined at speeds up to twice those for aluminium alloys and ten times those for steel, for a given power input.

The first alloys of magnesium contained aluminium and zine, but their high cost and low corrosion resistance prevented any real progress being made in their use. In 1925, Boyer discovered that the addition of an

excess quantity of manganese to molten magnesium not only precipitated impurities such as iron, but also greatly increased the corrosion resistance of the resultant alloy. Up to World War II, the principal magnesium alloys were to be found in the magnesium-manganese and magnesium-aluminium-zinc-manganese systems, and although German workers in 1938 discovered that zirconium had a potential grain refining effect on magnesium, it was left to M.E.L. to solve the problem of its introduction into the metal as a commercial operation. The discovery was timely since it accelerated progress in jet engine design, which depended on the availability of light heat-resistant metals. Additions of rare-earth metals to the binary and ternary zirconium-containing alloys were studied, and materials developed which had greatly improved properties at elevated temperatures. Whereas the classical alloys cannot be used above 100° C., a casting alloy containing zirconium, rare-earth metals and zinc (ZREI) is practicable at 250° C., and later developments based on the magnesium-zirconiumthorium-zinc system have resulted in ZT1, an alloy which can be used at temperatures up to 350° C. Attention is currently being paid to the development of wrought magnesium alloys containing thorium for use at elevated temperatures.

Applications

In the early days, magnesium suffered from prejudices of inflammability and corrodibility. In actual fact, the high thermal conductivity of magnesium prevents it from burning unless it is first melted by an otherwise disastrous fire. Reference has already been made to the improvement in corrosion resistance effected by the addition of manganese. Furthermore, the development of very effective protective coatings enables magnesium to be used with complete confidence for all applications except those involving continuous immersion in sea water.

World War II played a big part in the expansion of the use of magnesium which has been a feature of the post-war years, because fabricators to whom magnesium



Pouring a casting in the sand foundry. During this operation the metal is subjected to sulphur dusting to inhibit oxidation.

alloys were previously unknown were trained to use them for war production, and became aware of their potentialities. The performance of these materials in service has given increased confidence in them to engineers and constructors, and many fabricators throughout the world are now spending money to advance the technology of magnesium alloys, and no longer depend solely on the efforts of the producers.

Outstanding applications of magnesium alloys include their widespread use in all countries for aircraft landing wheels, of which approximately a million were cast for British aviation alone during the war. They stand up well to the service conditions, as do the 52 lb. magnesium alloy transmission cases of the Standard Ferguson tractor. The latter is a highly stressed casting, being the backbone of the tractor, and despite the rough usage and adverse conditions to which it has been exposed, there have been no known failures. In German vehicle manufacture, too, magnesium is playing an important role. The 36 lb. of finished magnesium castings in the Volkswagen accounted for an estimated 11,000 tons of magnesium in 1955, probably the greatest single use of the metal in the world.

The rare-earth- and thorium-containing alloys are widely used in jet and propeller turbine engines, and several aircraft incorporate magnesium castings in their airframes. Welded sheet and extrusions are used in the internal structure of the Britannia, and the Westland S55 helicopter fuselage is skinned entirely in magnesium alloy sheet.

Magnesium alloys are used in the engines of commercial vehicles, and in portable power tools, chain saws, textile machinery, vacuum cleaners, typewriters, cameras, binoculars, etc., and thick sheet is now being used in large quantities for heavy vehicle bodies. Reference should also be made to the use of magnesium alloys as a canning material for the fuel element in gascooled nuclear energy reactors. Non-engineering applications of magnesium include its use in the extraction of titanium by the Kroll process, as an alloying element in aluminium alloys, and as a material for making line

blocks in the printing industry, where its admirable etching qualities are advantageous.

The high contact potential of magnesium and its freedom from polarisation effects make it an ideal material to employ as galvanic anodes for the cathodic protection of corrodible metals, particularly iron and steel. Many thousands of tons of such anodes have been used to protect pipelines, buried structures, and the hulls and compartments of ships, particularly oil tankers.

Future Prospects

Progress in the application of magnesium and its alloys in the past ten years has been very rapid, particu-

larly in the U.S.A., where fabricators are confident of an assured and expanding usage, and are working on developments to find new and wider markets for their products. Confidence in the future was also expressed by Major C. J. P. Ball, chairman of Magnesium Elektron, Ltd., in concluding his Presidential Address to the Institute of Metals last March, when he said: "After more than thirty years experience of magnesium, I remain more convinced than ever that, as the inherent advantages of this lightest of metals become more widely known, the demand will rapidly increase, extraction and fabricating costs will be lowered, alloys will be further improved, and the future progress of magnesium will parallel or even improve upon that of aluminium."

French West African Alumina Project

LUMINIUM, LTD., is planning an initial investment of some \$100 million in a wholly-owned French subsidiary for the creation of a new bauxite and alumina industry in French West Africa to serve export markets. A joint statement issued by Aluminium, Ltd., in Montreal, and the subsidiary, Bauxites du Midi, in Paris, says that, following intensive engineering studies, plans have now been formulated for the creation of an important bauxite and alumina exporting industry in French Guinea. These plans are based on the development of the large bauxite deposits to be found in the territory, deposits which assume a particular importance in view of the continuous growth of world aluminium production. Bauxites du Midi some time ago commenced taking steps with the appropriate French and Territorial authorities to obtain all the approvals and guarantees it will require before moving on to the actual construction

The plans envisage the creation of a new industrial complex in French West Africa, about 345 miles South East of Dakar and 185 miles North West of Conakry, in an undeveloped area of French Guinea within the Boké region. The realization of these plans will be undertaken by Bauxites du Midi, which is already the bauxite mining pioneer of French West Africa. In 1952 the company commenced operations for the mining, beneficiation and shipment of this mineral from the Los Islands, off the mainland at Conakry in French Guinea. However, this undertaking was concerned only with bauxite production, which is the first step in the production of aluminium. From this bauxite, alumina (the oxide of aluminium) must first be extracted by means of complicated chemical and other processes which eliminate the other constituents of bauxite. The alumina is then reduced to aluminium metal by electrolysis.

On broad lines the plans involve :-

- (a) the development of new bauxite sources in the North West area of Guinea;
- (b) the construction of about 75 miles of railroad to the Atlantic coast;
- (c) the establishment of new storage and port facilities at the mouth of the Rio Nunez;
- (d) the construction of a chemical works with a capacity of 250,000 short tons of alumina per year; and
- (e) the construction of town-site facilities for employees and staff.

In order to realize the first step of the development under which the company will provide the port, railroad and town-site installations, as well as the industrial plants, it is estimated that an investment of the order of £100 million will be required over the construction period of about five years. The plans will provide a foundation on which could be built a capacity many times the initial production, as and when it is needed.

Entirely independent of the alumina project, Aluminium, Ltd., agreed last March to participate in the establishment of an aluminium industry in Guinea. The creation of the aluminium industry based on the hydroelectric potential of the Konkouré River is now being studied by Péchiney-Ugine, the principal French producers of alumina and aluminium, in collaboration with Aluminium, Ltd., and a group of European producers. It is still too early to determine the tonnage of alumina which the new plant of Bauxites du Midi will supply to the future aluminium works.

This new project is a natural consequence of a general long-term programme as well as of activities already undertaken in French West Africa by Aluminium, Ltd., and Bauxites du Midi since well before the last World War. The first stage of the programme involved the establishment of mining, milling and shipping facilities in the Los Islands, capable of producing and exporting a minimum of 250,000 short tons of beneficiated bauxite per annum. This stage was completed in 1952 at a cost of about \$12 million, and for the past two years shipments from these plants to North American and European markets have been increased to about 500,000 short tons per annum.

The second stage involved a similar enterprise on the mainland, in the Boké region, also in French Guinea. About \$2 millions have been expended since 1946 on exploration and prospecting in the region. These activities have confirmed the existence of bauxite resources of sufficient importance to justify the preparation of plans, not only for a bauxite industry, but also for an alumina industry to process an important part of the bauxite into alumina in the territory. The investment of \$100 million will, in itself, be a stimulant of the first order to the economic development of French Guinea.

If the various steps and formalities can be brought to a satisfactory conclusion, the construction of this whole industry should start in 1957. The first ton of bauxite could be shipped in 1960 and the first ton of alumina in 1961.

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NEWS AND ANNOUNCEMENTS

Research Techniques Course

ALWEEKLY series of lectures on Research Techniques in Metallurgy has been organised by the Metallurgy Department of the Battersea Polytechnic. The first lecture will be given on January 8th, 1957, and the series will terminate on April 2nd. The subjects to be discussed, in chronological order, are as follows: "Library Research" (Mr. R. F. EATWELL); "Materials of Research" (Dr. M. J. Olney); "Techniques of High Vacuum" (Dr. A. S. Baxter); "Topography and Structure of Metal Surfaces" (Dr. D. Tabor); "Electrolytic Polishing and Etching" (Dr. M. Colle);
"Mechanical Methods of Specimen Preparation" (Mr. R. E. LISMER); "Techniques of Optical Microscopy (Dr. J. A. Belk); "Electron Metallography" (Dr. J. Nutting); "Electron Diffraction Techniques" (Dr. J. W. Menter); "X-Ray Techniques in Metallurgy" (Dr. K. Robinson); "Single Crystals in Metallurgical Research" (Prof. R. W. K. Honeycombe); "Attainment and Measurement of High Temperatures in the Laboratory " (Mr. A. E. S. White); and "Miscellaneous Techniques in Metallurgical Research" (Dr. J. S. L.

The fee for the Course is 30s. and application forms should be obtained from the Secretary (Research Techniques Course), Battersea Polytechnic, Battersea

Park Road, London, S.W.11.

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Thermodynamics Course for Metallurgists

A Course on Thermodynamics for Metallurgists will be held in the Department of Metallurgy, St. George's Square, Sheffield, 1, from January 14th to February 16th, 1957, inclusive, and will comprise lectures, discussions and practical work.

Applications are now invited, and should be made as soon as possible to: The Professor of Metallurgy, The University of Sheffield, St. George's Square, Sheffield, 1. The fee for the course of five weeks'

duration is £21.

This course will assume no previous knowledge of thermodynamics, and will cover topics grouped under the following headings: the fundamentals of thermodynamics; thermodynamic functions; experimental application to phase diagrams; other applications; thermodynamics of extraction metallurgy: and thermodynamics of refractory systems.

Nuclear Energy Trade Associations' Conference

The practical development of nuclear energy engineering techniques is being taken in its stride by manufacturers. These lie in different sectors of the engineering industry for which separate trade associations serve. In order to establish a permanent means of liaison on matters relating to nuclear energy and associated developments, the Nuclear Energy Trade Associations' Conference (N.F.T.A.C.), has been formed. Membership, which in no way restricts the autonomy of the participants, is

open to those trade associations concerned with nuclear energy equipment.

The founder members of N.E.T.A.C., all of which are closely connected with the field of nuclear energy, are: the British Chemical Plant Manufacturers' Association; the British Electrical and Allied Manufacturers' Association; the British Engineers' Association; the Scientific Instrument Manufacturers' Association; and the Water-Tube Boilermakers' Association.

N.E.T.A.C. has already initiated action to make better known overseas the achievements of British industry in the nuclear engineering field. In this task N.E.T.A.C. is advised by a Publicity Committee on which experts from among the leading British manufacturers and civil engineering contractors concerned are serving. The address of the Nuclear Energy Trade Associations' Conference is that of the British Engineers' Association (whose staff serve as Secretariat), 32 Victoria Street, London, S.W.1.

Institute of Metals Awards

THE following awards are announced by the Institute of Metals:

The Institute of Metals (Platinum) Medal for 1957.— To Dr. Maurice Cook, Joint Managing Director, Imperial Chemical Industries, Ltd., Metals Division, in recognition of his outstanding contributions to the science of metallurgy, to the non-ferrous metals industry, and to the welfare of the metallurgical profession.

The Rosenhain Medal for 1957 .- To Dr. H. K. HARDY, Research Manager, United Kingdom Atomic Energy Authority, Industrial Group, in recognition of his outstanding contributions to knowledge in the field of physical metallurgy.

Iron and Steel Prices Increased

THE Iron and Steel Board has announced increases in the maximum prices of iron and steel products which became effective on December 17th, 1956. The industry has carried increased costs in the way of inland transport, coal and wages since the Spring, and later in the year (before the closing of the Suez Canal) there was a further increase in the landed costs of imported goods. Although it has been possible for the industry to offset these increases to some degree by increased efficiency, the net burden remains substantial. The board considers the price increases necessary in view of these rising costs and the need to support the heavy capital investment for the continuing expansion of the industry.

Gas in Industry Exhibition

EVERY phase of the application of gas in industry is to be displayed at the Royal Horticultural Hall, London, from January 22nd to February 2nd, 1957. This exhibition, "Gas at Work in Industry," will be the first of its kind. Organised by four Area Gas Boards—North Thames, South Eastern, Southern and Eastern-it will consist of working models and demonstrations of the use

of gas in industry. Its main purpose is to show the large variety of modern processes in which gas plays an important and often an essential part. The appliances and products of over 70 manufacturers will be represented, and exhibits on show will range from gas fired water and space heaters to highly complex items of equipment for shell moulding, tool cutting, flame hardening, and heat treatment.

Packaging Exhibition

PACKAGING must rank for top executive study amongst manufacturers of consumable goods, for it is computed that between 50% and 75% of their factory space, labour and money is expended on packaging. Advance information indicates that the Packaging Exhibition, to be held at Olympia from January 22nd to February 1st, 1957, will not only be the biggest show ever, but will offer the opportunity of obtaining expert advice from more than 200 manufacturers of packaging plant and materials. Concurrently with the Exhibition a Packaging Convention has been organised by The Institute of Packaging, the theme of which is "Better Packaging Means Better Living."

South African Coking Plant Order

Woodall-Duckham Construction Co., Ltd., announce that the South African Iron & Steel Industrial Corporation, Ltd., has just ordered another coke oven installation valued at approximately £2m. for their Vanderbijl Park Works. This is the ninth battery of W-D Becker coke ovens to be ordered by ISCOR and the fourth within the last two years. It comprises 55 ovens as does the battery now nearing completion at Vanderbijl. Two further batteries of 51 ovens are also under construction at the Pretoria Works. The total value of these four contracts approaches £6m.

ISCOR's continuing development is both an indication of and a vital contribution to the rapid industrial expansion which is taking place in South Africa. All the refractories and a major proportion of the steelwork and ironwork, including the oven machines for the new coking plant, will be fabricated in that country. In addition to the battery and machines, the new contract includes a complete by-product recovery plant, coal blending and handling and coke handling plants, and civil engineering work. When this plant is completed, the Vanderbijl Works will carbonise about 3,600 tons of coal daily.

Nickel Price

The Mond Nickel Co., Ltd., announces that, following the simultaneous announcements in Canada by The International Nickel Company of Canada, Ltd., of its project in Manitoba for opening two new nickel mines and of an increase in the price of nickel effective December 6th, it has, as from the same date, raised its price for refined nickel in the U.K. to £600 per ton delivered works, with appropriate increases for other countries.

B.S.C.R.A. Move

The British Steel Castings Research Association has moved from Broomsgrove Lodge to its new laboratories at East Bank Road, Sheffield, 2 (Tel.: Sheffield 28647).

Personal News

Mr. H. E. Dixon, has resigned his position as Chief Metallurgist to the British Welding Research Association in order to take up a senior appointment with Richardson Westgarth Atomic, Ltd. He is succeeded by Mr. P. T. HOULDCROFT.

The Power Gas Corporation, Ltd., recently announced the appointment of two Divisional Directors who will be responsible for the development of the Company's trade within special fields. Mr. P. M. K. Embling will be concerned with the Chemical Plant Division of The Power-Gas Corporation, Ltd., and Mr. D. R. Brown with the Blast Furnace Division of Ashmore, Benson, Pease & Co.

MISS E. HARBORNE has resigned from her position as Managing Director of Arthur Scrivener, Ltd., for health reasons. She will, however, retain her seat on the Board and continue to act in an executive capacity. Mr. W. V. HODGSON has been appointed Managing Director.

Mr. L. W. Stevens-Wilson has been appointed Development Officer to the British Iron and Steel Research Association in succession to Dr. T. E. Dancy, who resigned recently to take up an appointment in the United States.

METROPOLITAN-VICKERS ELECTRICAL Co., LTD., announces that as from January 1st, 1957, Mr. H. H. LUSTY, is appointed Manager, Publicity Department. He succeeds Mr. E. E. WALKER, who is retiring but will act in an advisory capacity until the end of January.

MR. R. DINGWALL, who has been works manager of the Company's Lackenby steelworks since 1953, has been appointed Deputy Chief Engineer of Dorman Long (Steel), Ltd., and MR. J. S. CURPHY, M.B.E., has been appointed Superintendent of all the Company's steel plants.

THE Council of the O.E.E.C. has appointed Dr. Alexander King, C.B.E., Deputy Director of the European Productivity Agency. He succeeds Mr. Edwin Fletcher, who has returned to the T.U.C., from which he was seconded in 1955.

MR. Tom Bishop is relinquishing his positions as Metallurgical Editor of *Iron and Coal Trades Review* and Editor of *Metal Treatment and Drop Forging* in January. He is joining the metallurgical staff of John Miles & Partners (London), Ltd., consulting engineers for metallurgical industries, of 76 Cannon Street, London, E.C.4. METAL INDUSTRIES, LTD., announces that DR. W. H. GARRETT has been appointed a Director of the Company. AMBER PHARMACEUTICALS, LTD., announce the appointment of MR. FRANCIS DE BUDA to the Board.

MR. R. W. Ruddle, who has been the Head of the Melting and Casting Section of the British Non-Ferrous Metals Research Association since 1947, is to join the Foundry Services Group on 1st February, 1957. His new appointment will be that of Technical Manager of Foundry Services Incorporated, Columbus, Ohio, U.S.A. BIRMINGHAM ALUMINIUM CASTING (1903) Co., Ltd. announce that Mr. R. V. Dowle has been appointed General Works Superintendent of the Company. Mr. Dowle, who has been associated with the Birmid Industries Group for 28 years, was previously Manager of the Aluminium Sand Foundry.

Mr. S. Parker has recently been appointed Applications Engineer by Radiovisor Parent, Ltd.

RECENT DEVELOPMENTS MATERIALS : PROCESSES : EQUIPMENT

Pressure Controllers

A NEW family of seven slack-diaphragm-operated pressure controllers is announced by George Kent, Ltd. These controllers are all of the non-indicating, non-recording, blind-setting type, and comprise suction or pressure controllers, including a special-purpose furnace-pressure controller; differential-pressure controllers; a special-purpose mill controller; and an air-flow controller or differential-pressure transmitter. The general appearance of all these instruments in the FAF group is similar, as shown in the illustration. The instrument door measures 18×15 in., and all the FAF instruments are suitable for wall, panel or post mounting, although normally the instruments are not mounted on a panel but near to the operating point.

The edgewise gauge at the upper centre of the door shows the "supply" and "control" air pressures of the Kent Mark 20 controlling circuit, expressed as percentages between minimum and maximum range. Any one of a number of mechanisms in the Kent Mark 20 series of pneumatic-controlling units can be fitted in the instruments, covering the following control terms and facilities: proportional only (0-60% proportional band); proportional (0-60%) + derivative; proportional (0-65%) + integral; and proportional (0-65%) + integral + derivative. The controlling units with 0-60% proportional band are fitted only where it is known that the band could in practice be 5% or less. This is because the FAF instruments are substantially of the force-balance type. The output signal of the Mark 20 controlling unit varies between 3 and 15 lb./sq. in. Controlling units employing integral and/or derivative action



FAF controller with door open.

are provided with control-action adjusting knobs scaled in time units between 0.15 and 40 minutes action time.

The FAF instrument case encloses an air-reducing valve, a relay, and an auto/manual switch. Where special testing and servicing facilities are required for the controlling unit, the instrument is associated with the Kent auto/manual/test/service and remote manual station. Where "overshoot" of the desired pressure value, following a period of regulator saturation, is likely to cause difficulty, and where integral action is used in the controlling unit, Kent make available their exclusive automatic bleed unit and/or automatic injector unit for insertion in the control circuit, connected up as an integral-pressure desaturator, within the FAF case.

All the FAF instruments are designed to operate with the diaphragms in the mid-position, corresponding to a 9 lb./sq. in. output of the 3–15 lb./sq. in. controlling unit. The desired-value setting of all the controllers is, of course, easily adjusted, on opening the door of the instrument. The only maintenance required by these FAF controllers in use is the occasional removal of condensation from the filter sump of the controlling unit.

Model FAF 1 is a special-purpose furnace-pressure controller, which can be set to control any pressure between $+\frac{1}{2}$ in. and $-\frac{1}{2}$ in. w.g. Usually one diaphragm side chamber is connected to the furnace combustion space and the other side chamber to the atmosphere at the furnace location. The atmosphere-piping connection follows the trail of the furnace-connection piping up to the point of entry into the furnace of the latter, thus compensating for any barometric-pressure changes and temperature changes in the connections to the controller. A deviation of $\pm \frac{1}{8}$ in. w.g. from the desired pressure value causes the instrument to move the plant regulator through its full travel.

The static suction or pressure controllers (FAF 2-7) are of two main types: (a) for values up to 6 in. w.g.; and (b) for values up to 48 in. w.g.

In the up-to-6 in. controllers, the ranges available are: 0 to \pm $1\frac{1}{4}$ in. w.g.; 0 to \pm 3 in. w.g.; and 0 to \pm 6 in. w.g. A typical application of the low-range pressure controller is for gas-offtake pressure-control duties in schemes for the automatic control of operation of gas producers. A subsidiary diaphragm chamber is employed in the up-to-48 in. w.g. controllers, where the ranges available are: 0 to \pm 12 in. w.g.; 0 to \pm 24 in. w.g.; and 0 to \pm 48 in. w.g.

The division of ranges for the differential-pressure controllers (FAF 02-07) is again up to 6 in. w.g. and up to 48 in. w.g., with sub-divisions (positive) as for the static-pressure controllers above. The maximum permissible static pressure is 2 lb./ sq. in.

The mill-load controller (FAF 001), 1 in.-13 in. w.g., is a special-purpose instrument, designed for the automatic load control of suction- or pressure-type pulverising mills. The maximum static pressure permissible with this instrument is again 2 lb./sq. in.

The air-flow controller or differential-pressure transmitter (FAF 002) is a dual-purpose instrument with a range of 1 in. w.g./15 lb./sq. in., the movement of the main slack diaphragm being modified by the operation

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of a bellows unit. It is used as an air-flow controller, the desired value of which can be set by an air pressure fed into the bellows unit. It is also applied as a forcebalance differential-pressure transmitter suitable for lowhead measurement, providing an air signal proportional to received differential pressure. The diaphragm casing is suitable for a maximum static pressure of 2 lb./sq. in.

George Kent, Ltd., Luton, Beds.

New Fluorescent Flaw Detection Ink

A NEW tool for detecting minute surface defects in nonmagnetisable materials-Britemor Fluorescent Flaw Detection Ink—has been developed by Manchester Oil Refinery, Ltd. The basis of the new method is, in fact, the old oil and chalk technique. In place of ordinary mineral oil, however, a special carrier medium has been substituted, which has not previously been used for this purpose, and which is loaded with a blend of fluorescent dyes that are claimed to give the most brilliant flaw indications yet achieved.

After application of the "ink" the component under test is rinsed with Quicmor wash, washed under cold water, and then dried either by wiping or with an air blast. The flaw pattern is then "developed" by dusting with a special Dry Powder Developer. Final examination is carried out under an ultra violet light source fitted with a Wood's glass filter. The extremely brilliant indications produced minimise operator fatigue.

Manchester Oil Refinery (Sales), Ltd., Twining Road. Trafford Park, Manchester, 18.

Capacity-Type Level Indicators and Controllers

FIELDEN ELECTRONICS, LTD., have now had five years' experience in the application to industry of capacitytype level indicators, and controllers, during which time a wide range of probes has been evolved to meet every conceivable problem. The original capacity-type level controller, the Tektor J.1, was produced in 1951, and the J.3, just released, is a direct development of this basic idea, incorporating all the knowledge and experience garnered in the industrial application of its two forerunners, the J.1 and J.2.

The unique reliability of the instrument has been achieved by its simple and robust design, which com-



Tektor Type J level controller

prises a simple oscillatory circuit which, when subject to a change of electrical capacity operates a relay for the control of electrical contactors, visible and audible alarms, etc. This change of capacity is brought about by the presence, or absence, of the material under measurement either liquid or solid—and this is initiated by a simple metal rod inserted in the container at the point at which control is required. This method eliminates any moving parts in the container.

The Tektor is offered in two standard forms, the type J.3, which is housed in a die-cast aluminium case, suitable for normal industrial applications, and the type J.5, housed in a weatherproof sheet metal case for more arduous industrial conditions. Flameproof models are also available. Although the continuous development of this instrument has taken several years, the J.3, its latest form, is believed to be the least expensive level

controller in the world.

The principle of capacitance has been extended to provide equipment for the continuous indication of level. The Telstor L.F.3 has been developed from earlier types, the L.F.1 and the L.F.2, and again represents the experience gained over a number of years. The capacity change is initiated by an electrode suspended from the top of the container and extending to its full depth. The contents of the container may then be displayed in indicators situated at a remote point from the actual plant, even up to half a mile away.

Fielden Electronics, Ltd., Paston Road, Wythenshawe, Manchester, 22.

Electronic Air Filter

THE Trion Electronic Air Filter which has recently been introduced into the U.K. is particularly suitable for use in industries where extremely clean air is required. It ensures air free of dust, dirt, smoke, bacteria and other airborne contaminents, and has obvious advantages in that it eliminates product contamination, reduces maintenance and cleaning costs and improves working conditions. The Trion filter, which utilises the principle of electrostatic precipitation, collects all airborne contaminents regardless of size. The smallest visible particle is about ten microns in diameter (1 micron = 0.001 mm.), yet it can be proved that particles less than 0.01 micron are removed; in this connection it is interesting to note that bacteria average approximately

All particles in the air receive a positive electric charge as they pass through a high voltage (13,000v. D.C.) ionising screen. These particles are then attracted and adhere to the collecting plates which form the negative elements of an electrostatic field set up between series of parallel plates, electrically charged alternately positive and negative by means of a 6,500v. D.C. supply. Periodically, depending on the dirt content of the air, the dirt must be removed from the collecting cell. This is accomplished by merely opening a valve which sprays the cell with water and washes the dirt into a drain. The ionising collecting cells, each of which is a complete unit, are rigidly constructed throughout of aluminium for corrosion resistance and ease of handling. The framework of the cell incorporates a venturi-type nozzle approach, which prevents by pass of uncleaned air, and permits the installation of insulators out of the air Fine tungsten ionising wires are suspended between tubular electrodes and mounted in front of a

series of vertical parallel aluminium plates, which are accurately positioned and rigidly held to uniform spacing. All ionising wires are the same size and

preformed for easy replacement.

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The Trion filter is essentially a unit for applications in which the total dust loading is substantially less than that occurring in heavy industrial plant, but which is sufficiently high to have deleterious effects. It is being marketed in the United Kingdom by W. C. Holmes & Co., Ltd., of Huddersfield and Harris Engineering Co., Ltd., of London. Holmes are handling industrial applications and Harris commercial applications.

W. C. Holmes & Co., Ltd., Turnbridge, Huddersfield.

Undergear for Mobile Welding Sets

Quasi-Arc, Ltd., announce the development of a 4-wheeled type Flexitor suspension undergear for attachment to standard 300 and 400 amp. diesel and petrol-driven welding sets and the double operator 300 amp. diesel set. The undergear consists of two main parts, the rear and front wheel assemblies, the latter incorporating a turntable with hinged towbar. Each part is designed for bolting direct to the welding set. The undergear has overrun and parking brakes which operate on the rear wheels only, and each wheel is independently suspended by means of a Flexitor suspension unit, which comprises a cylindrical shaft bonded to a rubber block which is also bonded to and contained by a flanged steel housing.

Flexitor suspension is made by the Bramber Engineering Co., Ltd., and is a most up-to-date method of suspension as there are no parts to wear, no traps for dust and dirt, and no lubrication is required. The undergear has been subjected to extensive tests, and gives smooth running for normal road towing. It is robust and is able to withstand the arduous conditions experienced on oil

sites and rough country locations.

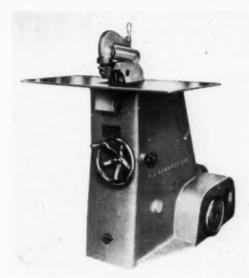
Quasi-Arc, Ltd., Bilston, Staffs.

Metal Forming and Flanging Machine

With appropriate forming tools, the Besco Model HS metal forming and flanging machine will perform a comprehensive range of operations on a wide variety of thicknesses and sheet metals, including wired or false wired edges on external and internal curved or straight edges, and plain, return bends, off-set, flared, large radii and half-round flanges, also on curved or straight edges.

Material up to a maximum thickness of 12 s.w.g. is formed or flanged by the oscillation of a short brakehead tool against a hold-down at a speed of approximately 213 or 426 strokes per minute, the total angle of this motion being controlled by a handwheel at the front of the machine. Whilst passing through the forming head, where the working components consist of a holddown, brakehead tool and an anvil, the material to be worked is supported by a flat table measuring 36 in. long × 21 in. deep and 36 in. from the floor. The brakehead tool throws up a flange, the maximum height of which is 17 in., and completes the operation against the holddown which grips the work to the anvil. Mounted on the solid frame of the machine, and flush with the top surface of the table, the anvil provides rigid support to the hold-down, which automatically releases the work after the brakehead stroke.

The 1 h.p. electric motor is situated below the operat-



Besco Model HS metal forming and flanging machine.

ing level, suitable for 400/440 volts, 3 phase, 50 cycles supply and has synchronous speeds of 1,500 and 750 r.p.m. An automatic direct switching starter and two-speed contactor, with built-in "fast," "slow" and "stop" push buttons, comprise the control gear. The machine is supplied with one complete set of tools for a given gauge or flange.

F. J. Edwards, Ltd., 359–361, Euston Road, London, N.W.1.

Thermostats

DEVELOPED particularly for the control of automatic fired boilers, the Sunvic T.Q.P. water thermostat and F.S.1 flue thermostat are also admirably suited for many and varied temperature control applications.

The T.Q.P. satisfies the need for a simple and robust control device providing a change-over switch facility, and has been designed for long service without attention. The head is contained in a two part cast box provided with a $\frac{3}{4}$ in. conduit entry. Adjustment of the temperature setting is by means of a small knurled knob on the top of the body casting. The scale is clearly marked in °F. and the range covered is 70–190° F., with a temperature differential of 7–12° F. The change-over contacts give ratings of 250 V., 15 A., A.C. on the front contact, 250 V., 5 A., A.C. on the back contact, and 250 V., 0·1 A., D.C. on both contacts.

The F.S.1 also features change-over contacts, which in this case are operated from a bimetal helix via a clutch. The use of the clutch ensures that the contacts change on a rise or fall of approximately 50° F. at any flue gas temperature. Like the T.Q.P., the head is contained in a two part cast box provided with $\frac{3}{4}$ in. conduit entry. The maximum operating temperature is 750° F., with a maximum permissible temperature of 1,000° F. and a differential of about 50° F. The switch contact ratings are 250 V., 0·25 A., A.C., and 250 V., 0·1 A., D.C.

Sunvic Controls, Ltd., 10, Essex Street, Strand, London, W.C.2.

CURRENT LITERATURE

Book Notices

MANGANESE STEEL

Published for Hadfields, Ltd., by Oliver and Boyd, Tweeddale Court, Edinburgh, 1: 128 pp., 42 graphs and drawings, and many half-tone illustrations. 18s. net.

Although the title of this book is as given above it could, more properly, have been given the title "Hadfield Manganese Steel," the name by which manganese steel is known throughout the world, because of the contributions it has made to the solution of many wearing problems in the engineering and mining industries at home and abroad, from the end of the Victorian era to the present time. The contributors include a wide cross-section of the staff of the Hadfield organisation, and they have undoubtedly provided an authoritative and up-to-date statement of present knowledge on manganese steel.

The book commences with a resumé of the historical background of the invention by Sir Robert Hadfield, 73 years ago, of manganese steel, which was the forerunner of a long list of alloy steels now in commercial use, and refers, briefly, to the work which led up to it. The fact that his experiments with steels having a manganese content in the range $2 \cdot 5 - 7 \cdot 5\%$ showed them to be extremely brittle did not deter him from making further experiments using a higher manganese content. It was from these experiments that the remarkable properties of manganese steel were discovered, his first patent covering the range of 7-20% manganese content.

It is probable that the brittleness encountered in his early experiments, was due to the high carbon content of the steel. Whatever the cause, his researches undoubtedly provided the basis of a very successful, tough alloy steel, which has since been used widely for its ability to withstand heavy wear. With the increase in manganese and high carbon he ultimately found that a steel with a content of 12% manganese had outstanding properties and the nominal composition of the steel he introduced commercially was 1.2% carbon and 12.5% manganese. This became known as Hadfield manganese steel, a name still applied to it. The characteristic properties of this steel are only attained after correct heat treatment, in which condition the normal type used for many engineering purposes, is austenitic, tough and ductile. It is soft, as determined by indentation tests, but becomes hard and wear-resistant when workhardened

The various chapters in this book deal with the general characteristics of manganese steel; its manufacture and processing; properties; heat treatment; transformational characteristics and metallography; work-hardening, abrasion and wear resistance; influence of temperature on properties; modifications in composition; non-magnetic steel for electrical applications; machining; welding, hard-surfacing, brazing and oxygen cutting; and industrial applications. In the latter section, some typical examples are excellently illustrated.

It is a remarkable fact that Hadfield's original method of producing manganese steel, by the addition of molten ferro-manganese to essentially carbon-free blown iron, using blown iron from either bottom-blown or sideblown convertors, is still the most economical and the most widely used process for the production of manganese steel for castings.

This book is both interesting and informative, and should prove an essential work of reference for engineers and metallurgists, and also a useful work for students. It carries a comprehensive bibliography and a useful index, and it is well produced.

LONDON INTERNATIONAL PRESSURE DIE CASTING CONFERENCE PROCEEDINGS

128 pp., numerous illustrations. Published by the Zinc Development Association, 34, Berkeley Square, London, W.1. 7s. 6d.

In October 1954, the European Pressure Die Casting Committee, with the help of the Zinc Development Association, held in London the first international conference ever to be devoted solely to pressure die casting. 150 delegates from 13 countries attended and freely exchanged information and ideas on various aspects of the industry. The texts of these discussions and the papers presented, after careful editing in collaboration with the authors and speakers, have now been published as an official report. The delay in publication was caused by the removal of the Zinc Development Association from Oxford to London during 1955.

In a paper on "Desirable Trends in Die Casting Machine Development," H. R. Haag, a director of Dyson & Co., Enfield (1919), Ltd., describes the types of machines available and suggests future developments in their design. For example, a separately arranged die locking device is required. It could be pneumatically or hydraulically operated, and should provide for automatic compensation for thermal expansion, balanced or unbalanced, in die and machine members. It should also be capable of rapid adjustment for varying die height requirements and should react directly against the machine frame or the tie bars. Other subjects discussed include core pulling, cycle controls, and ease of setting and operating.

H. Patin, of Fonderie de Précision de Nanterre, draws some interesting comparisons between die casting and machining, pressing, and plastic moulding, and considers some of the factors to be examined before choosing the process best suited to manufacture a particular part. It is emphasised that early consultation between designer and die caster is essential, if the maximum benefit is to be obtained from the die casting process.

Three papers deal with the production, testing, and finishing of die castings. F. G. Woollard, a past Chairman of the Zine Alloy Die Casters' Association, and well known for his work on production methods, discusses the great advantages of pressure die casting as an aid to higher productivity, and examines means for increasing still further the market for die castings. The accompanying illustrations show the wide range of parts already being produced. G. Lieby, of Mahle-Werk G.m.b.H. describes in detail the technical testing of pressure die castings, including a random test plan. Examples of testing equipment and procedures are shown. A paper on "Finishes for Die Castings," by

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A. P. Fenn and L. A. J. Lodder, describes and compares the finishes available for die castings in zinc, aluminium and magnesium. Finally, there is a short account of the O.E.E.C. Tecaid Mission to America in 1954, whose report has since been published.

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Trade Publications

Two leaflets recently received from The Magnetic Equipment Co., Ltd., deal with vibrators and their control. Damp and sticky materials tend to bridge in hoppers and clog. To overcome this difficulty, the Magco bin vibrators described in leaflet No. 101 have been developed: they can also be fitted to tables for consolidating moulds and packages. Leaflet No. 102 deals with vibratory control gear.

THE aim of a new quarterly magazine, Arteries of Industry, published by the Compoflex Co., Ltd., as a development of their Flexibles Advisory Service, is to present a magazine that will appeal, by its interesting and entertaining articles, to as broad a field as possible, and not confine the readership to users and potential users of flexibles. The contents will include up-to-date information on the manufacture and use of flexible tubes and hoses, and details, as they become available, will be given of new developments. Some of the problems presented to, and solved by, the advisory service, will be printed in each issue, with the idea that they may help others faced with similar problems. Applications for inclusion on the mailing list will be welcomed by the company at 23-25, Northumberland Avenue, London, W.C.2.

The English Electric Company has issued a new 26-page publication describing its extensive range of products for A.C. are welding. Besides giving brief descriptions, ratings and characteristics for the single and multi-operator equipments, electrodes, and accessory equipment, sections are devoted to installation and maintenance, and to some welding problems and their possible cures.

We have received from British Furnaces, Ltd., Chesterfield, reprints of advertisements dealing with RX Atmosphere Generators and the Allcase Furnace. The former produce a uniform atmosphere for dry cyaniding gas carburizing, homogeneous carburizing or carbon restoration heat treatment, and for protecting surfaces during bright annealing and clean hardening processes. The Allcase Furnace is a batch type radiant tube heated unit with recirculating fan and enclosed quench. All controlled atmosphere steel treatments can be carried out with or without liquid quenching.

A LEAFLET recently issued by Hadfields, Ltd., gives particulars of arc welding electrodes and welding rods for hard-surfacing and reclamation. These are of solid drawn or tubular type and are divided into three groups suitable for metal arc welding, automatic electric welding and exy-acetylene welding.

The application of preheated blast to cupola furnaces in iron pundries has only been developed on a practical scale in recent years, due to the previous absence of reliable recuperators able to withstand the relatively high air temperatures and pressures concerned. The savings and penefits resulting from the installation of recuperative but blast equipment are claimed to be sufficient to

outweigh the capital cost, and a new booklet entitled "Hot Blast Cupola Plants" has recently been issued by Cupodel, Ltd., which describes a typical installation and discusses the effect of hot blast on the metallurgy and economics of iron melting.

The ninth of the series of blotters issued by A.P.V.-Paramount, Ltd., deals with Paralloy 3LS stainless steel castings. This is a free-machining, non-magnetic 18/13/3% chromium-nickel-molybdenum, austenitic stainless steel, having corrosion resisting properties superior to the straight 14 and 18% chromium compositions.

We have received from Alar, Ltd., an individual data sheet for LM-24, the aluminium-silicon-copper pressure diecasting alloy which was introduced into B.S.1490 during the revision last year. It has excellent casting characteristics and is generally a little simpler to pressure diecast than the higher silicon-containing alloys, except where the highest possible fluidity is required. The alloy also has good gravity diecasting characteristics, although it is not commonly used for chill castings.

OF two publications recently issued by George Kent Ltd., one, Publication No. 334, deals with the Hammel-Dahl Diaphragm-Operated Control Valve. This unit serves to translate the pneumatic signal emitted by the Kent Mark 20 controlling unit into a valve-opening position of the throttle valve, which is installed in the controlled line. Publication No. 2020 deals with the instrumentation of blast furnace plants and includes blower house instruments, store control instruments, furnace platform instruments, skip charging instruments, blast furnace gas cleaning plant instruments, fuel control office instruments and sinter plant instruments.

WE have received from Stein & Atkinson, Ltd., a catalogue illustrating the principal types of melting furnaces designed and constructed by the company, covering a wide range of requirements for both ferrous and non-ferrous materials. They include rotary furnaces, end-tilting rotary furnaces, lip axis pouring rotary furnaces and fixed hearth lip axis pouring furnaces.

The amount of technical information and data relating to the very wide range of tubular products made by Accles & Pollock, Ltd., is now so great that the company has decided to embrace it all in one comprehensive publication. This has recently been issued in a book which should prove a handy work of reference for designers and users of tubes and tubular parts. The various sections deal with range of products, tolerances, manufacturing practice, analyses and properties, British Specifications, American Specifications, notes on stainless steel, non-ferrous metals, relative corrodibility of metals, properties of carbon steel tubes, tapering rolls and Dayton tools and engineers' tables.

"MILLING CUTTERS," a 16 mm. colour sound film, which runs for approximately 30 minutes, is the third in the series presented by Murex, Ltd., dealing with the subject of cemented tungsten carbide. The opening sequences show the cutting action of some single-edge and multi-edge cutting tools, and each type of cutter is shown in operation in various works. Some time is then spent in dealing with the important subject of servicing the tools, and the concluding shots form a summary of the cutters seen throughout the film in the light of the main field of application of each.

A working model of the Tornado shot cleaning plant attracted considerable attention at the recent Fuel Efficiency Exhibition at Olympia. This plant, which is designed for cleaning heat exchanger surfaces, is made by Keith Blackman, Ltd., under licence from AB. A. Ekstroms Maskinaffar, Sweden, and is described in Keith Blackman's Publication No. 35. The leaflet discusses the method of operation and the advantages of the process, which include the fact that it can be operated without the slightest alteration in load.

WE have received from Kodak, Ltd., an Industrial X-Ray Catalogue which gives particulars of films, chemicals, exposure equipment, processing equipment and viewing equipment. The price list is available in

the form of a separate supplement.

CALMET chromium-nickel alloy steel, which is the subject of a new publication of The Calorizing Corporation of Great Britain, Ltd., has been developed for application in various industries where high temperature conditions prevail. Besides illustrations showing typical applications, the brochure includes particulars of the scaling characteristic and mechanical properties at room and elevated temperatures.

A NEW brochure devoted to their mining pumps has been produced by Megator Pumps and Compressors, Ltd. It covers the range of Megator M type snore pumps for face drainage, dust suppression and wet cutting, and of the larger GH type pumps with capacities up to 160 g.p.m. and heads up to 350 ft. The characteristics of these sliding shoe pumps are discussed and each type is fully described. Supporting tables give comprehensive details of the output of each pump and the relative

weights and dimensions.

PUBLISHED by Rem-Cru Titanium, Inc., Midland, Pa., U.S.A., to assist in the application and fabrication of titanium alloys, the October, 1956, issue of Rem-Cru Titanium Review contains notes on two jet engine applications of the metal, one in the combustion system and the other for compressor blades. The corrosion resistance of titanium is the reason for its choice in a cooler for sodium hypochlorite solution and as wire cloth for use in filtering assemblies, etc., both of which are referred to in this issue. Other features deal with the replacement of steel by titanium for outboard wing slat tracks in the Douglas A3D Sky Warrior, and the ductility of titanium alloys at very low temperatures.

The Mond Nickel Co., Ltd., has recently issued a new publication entitled "Nickel Plating for Engineers," which has been written primarily for engineers, designers and others who are interested in the possibilities which plating offers in design. No attempt has been made to cover plating procedures in great detail, but practices have been dealt with in so far as they affect the properties of the resultant deposits. The text includes a description of surface preparation in general, and for zinc-base alloys, aluminium and nickel-chromium-iron alloys in particular. Commonly used solutions, relevant plating procedures to maintain high quality work, plant required mechanical properties of deposits and methods of testing deposits are all dealt with in a highly compressed text. A good deal of information is given in the form of graphs or tables for quick reference.

In view of the widespread interest in automation, a film has been produced, showing automatic plants for electroplating and other metal finishing processes in actual operation on production. The 16 mm. film has a running time of approximately 10 minutes, and is available on loan from Silvercrown, Ltd., 178 Goswell Road, London, E.C.I.

We have received from Birlec, Ltd., copies of the new editions of their Publications 76 (Continuous Conveyor Furnaces) and 65 (Birlec Detroit Rocking Arc Electric Melting Furnace). Publication 76 has recently been revised to include photographs of a new annealing furnace for silicon steel, and of the gas carburising plant at E.N.V. Engineering Co., Ltd., Willesden. It also incorporates photographs showing a typical battery of shaker hearth conveyor furnaces feeding a shaker hearth conveyor system, and, finally, a photograph of the new automatic wire annealing furnace at Enfield Cables, Ltd.

Books Received

"Order-Disorder Phenomena." By Dr. E. W. Elcock. 166 pp. London and New York, 1956. Methuen and Co., Ltd., and John Wiley and Sons, Inc. 11s. 6d. net.

"Molybdenum." By Dr. L. Northcott. 222 pp. London, 1956. Butterworths Scientific Publications. 40s. Postage 1s. 3d. extra. The American edition of this book is published by Academic Press Inc.

"Thermochemical Data of Alloys." By O. Kubaschewski and J. Catterall. 200 pp. London and New York, 1956. Pergamon Press, Ltd. 45s. net.

"Grundlagen der Metallkunde in anschaulicher Darstellung." By Georg Masing. 153 pp., inc. 140 illustrations. Berlin/Gottingen/Heidelberg, 1955. Springer Verlag. Ladenpreis: DM 12·60.

"Alloy Series in Physical Metallurgy." By M. C. Smith. 338 pp. New York and London, 1956. Harper & Brothers and Constable & Co., Ltd. 50s. net.

"Analysis of Deformation." Vol. III. "Fluidity." By K. Swainger. 266 pp. inc. glossary and index. London, 1956. Chapman & Hall, Ltd. 65s. net.

"Metallurgical Analysis by means of the Spekker Photoelectric Absorptiometer." Second Edition. By F. W. Haywood and A.A.R. Wood. 292 pp. London, 1956. Hilger & Watts, Ltd. 40s. net.

"Metallurgy and Fuels." Vol. I. Edited by H. M. Finniston and J. P. Howe. Progress in Nuclear Energy, Series V. 805 pp. inc. index and numerous illustrations. London, 1956. Pergamon Press, Ltd. U.S.A. edition published by McGraw-Hill Book Co., Inc. 147s. net.

"Elements of X-ray Diffraction." By B. D. Cullity. 514 pp. inc. index and numerous illustrations. Massachusetts, 1956. Addison-Wesley Publishing Co., Inc.

British Agent: Academic Books, Ltd. 80s.

"The Electrolytic and Chemical Polishing of Metals in Research and Industry." By W. J. McG. Tegart. 129 pp. London, 1956. Pergamon Press, Ltd. 30s. net.

"Oxygen in Iron and Steel Making." By J. A. Charles, W. J. B. Chater and J. L. Harrison. 309 pp. inc. author and subject indexes and numerous illustrations. London, 1956. Butterworths Scientific Publications. 42s. By post 1s. 10d. extra.

"Laboratory Administration." By E. S. Hiscocks. 392 pp. London and New York, 1956. Macmillan &

Co., Ltd., and St. Martin's Press. 36s. net.

"Automation: Its Purpose and Future." By M. Pyke. 191 pp. London, 1956. Hutchinson's Scientific and Technical Publications. 16s. net.

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC INSTRUMENTS AND MATERIALS

JANUARY, 1957.

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Vol. LV, No. 327

The Volumetric Determination of Antimony in Antimony-Lead Alloys

By E. G. Brown, A.M.C.T., F.R.I.C., I. P. Forshaw and T. J. Hayes, A.R.I.C.

Research Department, British Enka, Ltd., Aintree

A rapid volumetric method for the determination of up to 12% of antimony in hard lead is described. A mixture of acetic acid and hydrogen peroxide is used as solvent for the alloy and sulphur dioxide solution is used for the antimony to antimony 111 reduction. Potassium bromate is the final titrant. The procedure is suitable for routine assistants and one determination may be completed in under an hour. Precision and accuracy are very satisfactory.

HAMILTON¹ has shown that lead and sheathing alloys are rapidly attacked by 30% hydrogen peroxide, and this observation has been used by van der Meulen² and by Black³ in methods for the volumetric determination of antimony in such alloys, a mixture of hydrogen peroxide and acetic acid being utilised as the reagent for dissolution of the metal. The present paper details our method for the determination of antimony in antimony-lead alloys containing up to 12% of antimony, using an adaption of Black's procedure. Black only reported figures up to 1% of antimony.

Method

Reagents

All reagents should conform to recognised analytical specifications.

Glacial Acetic Acid (sp. gr. 1.05).

Hydrogen Peroxide (100 volume concentration).

Hydrochloric Acid (sp. gr. 1.18).

Sulphur Dioxide Solution.—Obtain liquid sulphur dioxide by inversion of a syphon and dilute with water to give an approximately 10% v/v solution.

Potassium Bromate Solution (0·1 N).—Dry some freshly powdered potassium bromate at 120° C. for 1 to 2 hours, cool and weigh out accurately 2·784 g. of the material. Make up to 1 litre in a volumetric flesk

1 ml. of this solution = 0.006088 g, of antimony. Methyl Red Solution.—A 0.02% solution in 60% v/v ethanol.

Procedure

Weigh out accurately into a 500-ml. conical flask the sample of alloy, preferably in the form of drillings, according to the following scheme:—

 $\begin{array}{ccc} \text{Nominal Antimony Content} & & \text{Weight of Sample} \\ & 3\% & & 3g. \\ & 6\% & & 1 \cdot 5g. \\ & 12\% & & 0 \cdot 75g. \end{array}$

Dissolve in a freshly prepared, premixed solution of 0 ml. of water, 10 ml. of hydrogen peroxide and 5 ml.

of acetic acid. When dissolution is complete, add 25 ml. of hydrochloric acid and place on a steam bath for 10 minutes, to decompose peroxides and halogen oxides. Add 50 ml. of a 10% v/v sulphur dioxide solution and digest on a steam bath for a further 15 minutes. Then add 50 ml. of water and boil gently for a minimum time of 15 minutes to drive off all sulphur dioxide.

Titrate the warm solution with standard potassium bromate solution, adding 3 drops of methyl red indicator near the end point, and allowing 30 seconds to elapse between successive increments of titrant, swirling vigorously the whole time. Take the end point as being when the methyl red indicator is just decolorised.

For accurate work, run a blank determination through the whole procedure, using AnalaR lead instead of the antimony-lead alloy. For routine purposes the blank may be omitted.

Calculation

The alloy contains.

 $0.6088 \frac{(a-b)}{w} \%$ of antimony

where a = titration of 0.1 N potassium bromate in millilitres.

b =blank titration of $0 \cdot 1$ N potassium bromate in millilitres.

w = weight of alloy in grams.

Express the result to the nearest 0.01%.

Results

The results obtained by adding varying aliquots of $0\cdot 1$ N antimony solution (as potassium antimonyl tartrate solution) to AnalaR lead and analysing for antimony by the complete proposed procedure are shown in Table I, in which all titrations are corrected for blank.

The overall standard deviation of the results quoted in Table I is 0·39 mg., corresponding to 0·016% absolute, calculated on the antimony basis. The data show satisfactory accuracy and precision for the method over the range covered; there is no statistical evidence that higher or lower amounts of antimony than those added are recovered by the procedure.

TABLE II.—DETERMINATION OF ANTIMONY IN COMMERCIAL ANTIMONY-LEAD ALLOY.

Lead (g).	Antimony (mg.)		Antimony (%)	
	Added	Found	Added	Found
2.5	30 · 44	30 · 44 30 · 44 30 · 44 30 · 63	1.218	1 · 218 1 · 218 1 · 218 1 · 222
2.5	60+88	61 · 60 60 · 88 61 · 13 60 · 83	2-436	2 · 465 2 · 436 2 · 445 2 · 433
2.5	121.8	121 · 9 121 · 8 122 · 1 122 · 1	4-872	4.873 4.872 4.884 4.884
2-5	152-2	152 · 4 152 · 8 152 · 9 153 · 0	4-088	6-096 6-099 6-110
9-5	305-6	305 · 0 305 · 0 305 · 0 305 · 5	12-22	12·20 12·20 12·20 12·22
5-0	152 - 8	152·6 152·6	3-056	3 · 052 3 · 052

Operator	Weight of Alloy (g.)	2-505 2-501	
1	2-9955 3-2912		
2	8 · 3004 3 · 5459		2·508 2·506
3	4-1304 4-3630		2-538 2-505
4	3-2731 3-5681		2·498 2·520
5	3-6269 3-8028		2·505 2·510
6	4-3237 3-2345		2.505 2.505
		Mean	2.508

Twelve determinations of antimony in a commercial antimony-lead alloy were made in duplicate, using the procedure, by each of six operators of varying degrees of skill. The results are shown in Table II, uncorrected for blank.

The overall standard deviation of the above results is 0.011% absolute. There is no statistical evidence that any of the six operators obtains more variable or higher or lower results than the others. There is no evidence that the weight of alloy taken for analysis affects the result, over the range covered.

As a further check, known amounts of standard antimony solution were added to weighed amounts of the sample shown in Table II (antimony content $2\cdot508\%$) and the total antimony determined according to the method. Results were as in Table III, from which it will be seen that the recovery of antimony is satisfactory. The data of Tables I, II and III show that the method is sufficiently precise and accurate.

Discussion

Effect of Variation of Experimental Conditions on the Procedure

As shown above, the procedure is capable of very satisfactory accuracy and precision. It is, however, imperative that the method be rigidly adhered to, particularly with regard to the stated times. In our opinion, any failures with this method are due to non-compliance with this basic requirement, both from the viewpoint of incomplete decomposition of hydrogen peroxide before addition of sulphur dioxide, and also incomplete evolution of sulphur dioxide on boiling. The ratio of sulphur dioxide to antimony is also very important.

Table IV shows some preliminary results obtained on a sample of antimony-lead alloy, and indicates the influence of variation of some conditions on the result (using sodium sulphite as reductant.)

On the basis of Table IV, conditions were standardised at 10 minutes decomposition time, the equivalent of 5 g. of available sulphur dioxide for reduction purposes, 15 minutes digestion on the steam bath, and 15 minutes boiling to drive off all sulphur dioxide. As shown under

"Results" these conditions were extremely satisfactory for alloys containing up to 12% antimony. Black used a "few" minutes decomposition time and the equivalent of $2 \cdot 5$ g. of available sulphur dioxide. In practice, 50 ml. of a 10% v/v sulphur dioxide solution provides somewhat more than 5 g. of available sulphur dioxide.

Use of Sulphur Dioxide as Reductant

An aqueous solution of sulphur dioxide is recommended for the reduction of antimony to antimony (instead of sodium sulphite crystals) because of the extremely low overall blank value associated with this reagent. This never amounted to more than 0.09 ml., and may be as low as 0.03 ml.; the overall blank using sodium sulphite may be as high as 0.20 ml.

Inhomogenity of Antimony-Lead Alloys

According to Benedetti-Pichler, alloys having a high antimony content are often heterogeneous: care in sampling is thus required. The results quoted in Table II were obtained on drillings taken at various points on an approximately 1 in. length of lead pipe.

Interferences in the Method

The interference of various metals with the procedure was not studied, as we were concerned solely with hard lead containing only antimony as an alloying agent.

Advantages of the Method

The principal advantage of the procedure is speed; one analysis can be completed in about 50 mins., and it is possible to carry out several simultaneous determinations. The older methods of dissolution of antimony-lead alloys are time-consuming and in the case of bromine and hydrochloric acid, unpleasant. It is surprising that the dissolution method using acetic acid and hydrogen peroxide has not become more widely used. For instance, the recent comprehensive paper on the bromatometric determination of antimony in lead- and tin-base alloys by Detmar and van der Velde⁵ still uses bromine and hydrochloric acid as a solvent, although these authors admit that dissolution may take many hours. In our opinion the use of an acetic acid-hydrogen peroxide mixture as a solvent for lead and antimony-lead alloys renders obsolete any other dissolution procedures.

The proposed method has now been satisfactorily used in our Control Laboratory for some considerable time. There is no doubt it could be adapted for greater amounts of antimony than 12% if required.

TABLE III.—RECOVERY OF ANTIMONY IN THE PRESENCE OF A COMMERCIAL ANTIMONY-LEAD ALLOY.

Weight of Alloy (g.)	Total Antimony (mg.)		Antimony Added (mg.	
weight of Alloy (g.)	Present	Found	Present	Found
2·7944 2·9938 3·8546 3·4276	100·5 136·0 188·0 207·8	100·2 135·8 187·7 207·2	30 · 44 60 · 88 91 · 32 121 · 8	30·13 60·70 91·00 121·2

Conclusions

- (1) Hydrogen peroxide-acetic acid mixture is currently the most suitable and speedy solvent for the dissolution of antimony-lead alloys.
- (2) Aqueous sulphur dioxide solution is recommended for the reduction of antimony solutions in the proposed method.
- (3) Antimony may be determined in hard lead containing up to 12% of antimony by final titration with potassium bromate solution, with satisfactory accuracy and precision, the standard deviation of a single determination being 0.011% absolute as antimony. Rigid standardisation of experimental conditions is necessary.

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(4) The procedure is much more rapid than previously

TABLE IV.—INFLUENCE OF VARIATION OF EXPERIMENTAL CONDITIONS ON DETERMINATION OF ANTIMONY IN A COMMERCIAL SAMPLE.

Time for Decomposition of Hydrogen Peroxide (min.)	Amount of 10% Sodium Sulphite (Na ₈ SO ₃) Solution (ml.)	Boiling Time (min.)	Antimony Found
?	25	15	2.39 2.42
0	30	15	0 0
1	30	15	2-34 2-11
5	50	15	2-45 2-45
10	50	15	2-50 2-50
10	75	15	2-51 2-52
10	50 30 50 50 75	18	2.52 2.59
10	100	15	2-52 2-52

described determinations; the analysis may be completed in under 1 hr. and several determinations may be performed simultaneously.

Acknowledgment

Our thanks are due to the Directors of British Enka, Ltd., for permission to publish this communication.

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 van der Meulen, J. H., Chem. Weckbl., 1949, **45**, 633.
 Black, R. M., Analysis, 1969, **75**, 166.
 Benedetti-Pichler, A. A., "Introduction to the Microtechnique of Inorganic Analysis," 1942, p. 237.
 Detmar, D. A., and van der Velde, W., Anal. Chim. Acta, 1956, **15**, 173.

New British Analytical Standards

METHODS FOR THE ANALYSIS OF IRON AND STEEL (B.S. 1121:1956)

PART 14: DETERMINATION OF COPPER IN IRON AND STEEL. PRICE 2s. Part 25: Determination of Vanadium in Iron, Steel and Ferro-Vanadium. Price 2s. 6d. Part 36: Absorptiometric Determination of Copper in Iron and Steel. Price 2s.

B.S. 1121: Part 14, "Determination of Copper in Iron and Steel," is a revision of the part first issued in 1949. It is applicable up to 6.5% of copper and sets out two methods. Method A, based on precipitation as sulphide with sodium thiosulphate, is suited to the determination of low copper contents in steels which are soluble in dilute sulphuric acid. Its use may be considerably extended by using the decomposition procedure given for Method B. This also permits its application in the presence of alloying amounts of vanadium, niobium, tantalum, titanium or tungsten. Method B, based on precipitation as thiocyanate, is applicable to all steels, and is generally more suitable for highly alloyed steels and steels of high copper content. It is less accurate than Method A in the low copper ranges, e.g., 0-10%. In both methods the determination is completed by liberating the iodine equivalent of the copper with potassium iodide and titrating with standardized sodium thiosulphate.

B.S. 1121: Part 25, 'Determination of Vanadium in Iron, Steel and Ferro-Vanadium,' is a revision of Parts 25 and 27 which were first issued in 1952. It lays down two methods, one of which is suitable for all classes of iron and steel, and the other for all grades of ferro-vanadium. In both methods vanadium is oxidised to the quinquivalent condition with potassium permanganate in cold solution. Excess of potassium permanganate is reduced with sodium nitrite, and sulphamic acid is used to destroy the excess of nitrite. Hydrofluoric acid is added and the oxidised vanadium is titrated with standard ferrous ammonium sulphate, using preoxidised sodium diphenylamine sulphonate as indicator.

B.S. 1121: Part 36, 'Absorptiometric Determination

of Copper in Iron and Steel,' is a new part and specifies a method for determining up to 1% of copper. The principle of the method is that copper in a phosphoric sulphuric solution of the sample is complexed with 2-21 diquinolyl and the resultant coloured compound extracted in amyl alcohol. The copper content is determined by measurement of the absorption and evaluation from a calibration graph. The method has been found satisfactory in the presence of cobalt, manganese, nitrate, tungsten and other elements.

Copies of these British Standards are available from the British Standards Institution, Sales Branch, 2 Park Street, London W.1.

Jubilee Foundation Lecture

To mark the 25th Anniversary of the opening of the main college building in Howard Street, the Rotherham College of Technology Jubilee Foundation Lecture was delivered in the College Assembly Hall by Mr. R. F. JACKSON, Operations Group Leader in the Reactor Division of the Atomic Energy Research Establishment at Harwell, who took as his subject: "Research Reactors in the Nuclear Power Programme."

During the last quarter of a century the College has held a leading position in the Rotherham, Sheffield and surrounding districts, and has made a valuable contribution to industry and commerce in that area. In carrying out extensive pioneer work in technical education it has established a high reputation, regionally and nationally, and during the last four years it has become the regional centre for South Yorkshire for powder metallurgy, furnace brickwork and sandwich courses in mechanical and electrical engineering.

Recent Advances in "Dead-Stop End Point" Titrimetry

By John T. Stock

Chemistry Department, University of Connecticut, Storrs, Conn., U.S.A.

Thirty years have elapsed since Foulk and Bawden introduced their simple and elegant "dead-stop end point" titrimetric technique. Nevertheless, numerous developments in the applications of this technique and the apparatus employed have occurred during the past four years. Recent studies have placed the "dead-stop" method on a sound theoretical basis and it is now firmly established as a form of amperometric titration.

THE detection of the end point of a titration as an abrupt change in the current flow between two similar electrodes immersed in the solution being titrated dates back to 1897. Little use was made of the technique, however, until its possibilities were indicated some thirty years later by Foulk and Bawden. Under the name "dead-stop end point," given to it by these workers, the technique has found numerous applications. Many of these have been summarised in two reviews the present article surveys the more recent developments in the practical and theoretical aspects.

Apparatus

The electrode system described by Freedman⁴ is shown in Fig. 1. Wire-form platinum electrodes AA, which dip into the solution to be titrated, are silversoldered to copper leads and then cast in glass tube B, using a cold-setting epoxy resin. Resistors R_1 and R_2 , of total resistance 1,500-2,000 ohms, and connected in series with switch C and $1 \cdot 5$ -volt cell D, permit a fixed e.m.f. to be applied to the electrodes. The current flowing through the well-stirred solution is indicated by galvanometer E. Two principal cases of end point indication then arise. In the first of these, the current falls abruptly in the immediate vicinity of the end point, and is then virtually unaffected by continuing the titration. In the second case, the end point is signalled by a sudden increase in current, and is sometimes referred to as a "reversed dead-stop." An alternative and very suggestive term is "kick off end point." The term "dead-stop end point" has, however, been used quite generally for both of these cases, and also for a third one in which the current passes through a minimum at the end point.

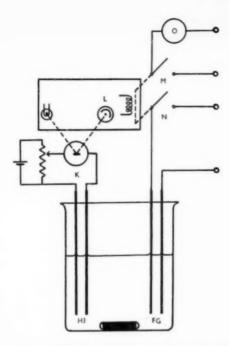
Using the comparatively high applied e.m.f. of 200 mV., Freedman4 found that the galvanometer could be replaced by a rugged 0-100 μ A. meter. In many cases, the applied e.m.f. may be only 10-15 mV., when a galvanometer of sensitivity 50-200 mm./μA. is needed. A microammeter may, however, be used in conjunction with a stable transistor device which amplifies the current six or seven times.6 In the titration of ferrous iron with normal potassium dichromate, the end point is so abrupt that no warning of approach is given with a normal dead-stop circuit. This difficulty may be overcome by a switching device which places the galvanometer in parallel with the electrodes during the greater part of the titration.7 A definite permanent increase in meter reading then occurs a little before the end point. The meter is now switched in series with the electrodes, and

Fig. 1.—Wire-form electrode system and circuit arrangement for dead-stop titrimetry.

the final drops of reagent are added slowly until a sudden decrease in the meter reading occurs.

An electronic dead-stop end point indicator was described well over twenty years ago, and apparatus of this type continues to appear.* In many cases a robust "magic eye" radio tuning indicator replaces the galvanometer. An interesting variation is an audible indicator.* The end point is indicated by a continuous note; transitory excess of reagent produces an intermittent note, so that adequate warning of end point approach is given.

The sharp change in current strength associated with an end point of the "dead-stop" type may be used to stop the titration automatically. This principle is particularly useful in coulometric titration, 10 in which the reagent is generated electrolytically. No burette is therefore required, since the amount of reagent is measured in terms of the quantity of electricity required to produce it. If the strength of the generation current is fixed, then the measurement reduces to one of time



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Fig. 2.—Schematic arrangement for automatic titration of mercaptans.

The automatic coulometric titrator shown schematically in Fig. 2 was developed for the determination of microgram quantities of mercaptans in petroleum stocks.11 Silver ions are generated by electrolysis at constant current between silver anode F and platinum cathode G. The electrolyte, to which the sample is added, is a mixture of ethanol and benzene containing ammonium nitrate and ammonia. Gold cathode H and platinum anode J comprise the detecting system. At the end point, excess silver ions increase the current flow between H and J, and hence the deflection of mirror galvanometer K. This activates phototube relay system L, which opens switches M and N. The constant generation current and the electrical timer O are thus simultaneously arrested. A run can be completed in The instrument has been licensed for 2-5 minutes. manufacture by the Central Scientific Co. of Chicago. Various other devices for arresting a titration at an amperometric end point have been described.12 A difficulty liable to be encountered with automatic systems is over-titration, arising principally from the lag in the indicator system. In a universally-applicable method described by Carson, 13 about 10% of the sample is withdrawn by means of a magnetically-operated syringe. The remainder is then titrated at a rapid rate to a preliminary end point, when the withdrawn portion is returned and the final end point is reached by slow titration.

Basic Principles

Although often classified in the past with potentiometric techniques, a dead-stop titration is carried out at fixed voltage, a greatly-increased flow of current indicating the end point.¹⁴ The titration is thus an amperometric (or, at least, amperoscopic) one, in which the current-strength changes so abruptly at the end point that there is no need to plot the titration curve. The dead-stop technique is therefore classified by Kolthoff⁵ as amperometric titration with two indicator electrodes.

Considerable advances in the theoretical aspects have been made during the past few years. Stressing the derivative-like nature of the dead-stop end point, Reilley, Cooke, and Furman developed a three-dimensional model for the interpretation and interconnection of electrometric processes. ¹⁵ A general theory of the dead-stop titration has been developed by Bradbury. ¹⁶ This theory is based upon the fact that, for a reversible electrode system, the current is controlled by the rates of diffusion of the reacting substances to and from the electrode. Bradbury developed a series of equations from which he was able to make useful predictions as to the accuracy and sensitivity of the method; he also considered irreversible systems.

Duyckaerts developed a general equation for the polarisation curve of an oxidation-reduction system, taking reaction-kinetics and concentration overpotential into account.17. He showed how this equation can be applied to titration curves obtainable in dead-stop titrations. A paper by Gauguin and Charlot, 18 published simultaneously with that of Duyckaerts, also considers polarisation curves, and establishes quantitatively the conditions for dead-stop titrimetry. Gauguin and Charlot point out that the "two-indicator electrode" amperometric titration curves obtained in an oxidationreduction reaction are of the three general types shown in Fig. 3. When the sample and titrant systems are equally reversible under the experimental conditions, the curve is of the general form (a). If the titrant system is the more reversible, a curve of form (b) will result. whereas curve (c) is associated with the greater reversibility of the sample system.

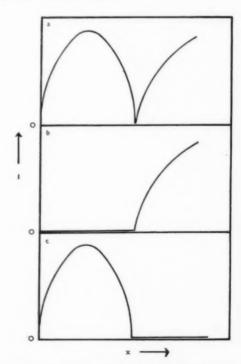


Fig. 3.—General forms of titration curves.

Following his earlier work in this field, Kies also deduced the form of the dead-stop titration curve for reversible cases. ¹⁹ Dealing with reversible or entirely irreversible systems, Kolthoff has discussed and classified potentiometric and amperometric titrations on the basis of voltammetry. ⁵ Pointing out that consistent terminology is desirable, Kolthoff defined an electrode as being polarised when it adopts a potential impressed upon it with little or no change of the current, and being ideally depolarised when upon passage of current the potential does not deviate from its reversible value. He developed a very simple mathematical treatment of the form of the amperometric titration curve obtained with two indicator electrodes.

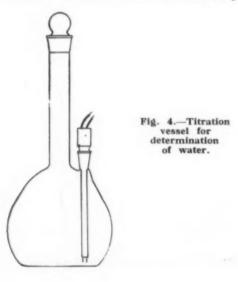
Determination of Water

More than thirteen years have passed since the deadstop end point technique was first shown to be applicable to the determination of water by titration with the Karl Fischer reagent.²⁰ However, the stream of publications in this important field shows no signs of diminishing. The reagent, which is a solution of sulphur dioxide, iodine, and pyridine in methanol, undergoes the following overall reaction with water:

$$\begin{split} \mathrm{SO_2} + \mathrm{l_2} + \mathrm{3C_5H_5N} + \mathrm{CH_3OH} + \mathrm{H_2O} = \\ \mathrm{C_5H_5NH.SO_4.CH_3} + \mathrm{2C_5H_5N.HI} \end{split}$$

Simple apparatus and quick preparation of the reagent are described by van Pelt and Keuker.²¹ Substitution of methyl Cellosolve for methanol in the formula for the Fischer reagent is reported to afford a substantial gain in stability.²² Other workers recommend the use of dioxan.²³

The titration cell recommended by Peters and Jungnickel²² is shown in Fig. 4. An interchangeable platinum electrode pair is inserted in a 250-ml. volumetric flask through a side-opening carrying a standard taper ground joint. The long narrow neck not only minimises ingress of atmospheric moisture, but also prevents collection of moisture on the burette tip when the latter is inserted in the neck. A somewhat more elaborate cell has been described by Campbell.²⁴ Menville and Henderson,²⁵ use an Erlenmeyer flask with sealed-in electrodes, closing the mouth of the flask with an amber rubber cap. The



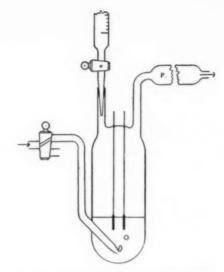


Fig. 5.—Flow-type cell for determination of moisture in gases.

latter has two small incisions, one of which permits insertion of the burette tip, while the other acts as a Bunsen valve. In the cell described by Praeger, ²⁶ the electrodes are placed one over the other and the sample is introduced through a side tube.

Applications of the electrometric Fischer titration are very diverse. The moisture content of sodium hydroxide may be determined by distillation with xylene and titration of the water entrained in the distillate.27 Small amounts of moisture in commercial sodium bicarbonate. causing lump-formation and poor flow, may be determined after extraction with dry methanol. A correction is applied for the small amount of bicarbonate which dissolves in the methanol.28 The determination of moisture in air, nitrogen, and hydrocarbon gases may be achieved by bubbling the gas through Fischer reagent contained in the flow-type cell shown in Fig. 5. Drying tube P prevents back-diffusion of moisture into the cell. Gas passed is measured by a meter connected to the exit.29 This flow technique has obvious possibilities for the examination of solid samples which are insoluble in, or react with, the Fischer reagent or an extracting liquid. An example is its application to the determination of absorbed moisture on uranium and uranium oxide.30

Direct determination of water in white fuming nitric acid may be determined after neutralisation with pyridine-dimethylformamide solution.31 To avoid violent reaction, the 1-2-gram sample is cautiously added to excess of the base. A slight excess of Fischer reagent is then added and the mixture back-titrated to a deadstop end point with a standard solution of water in methanol. Dead-stop Fischer titration has been used for the determination of water in medicinal tars,32 transformer oils, 33 propellant explosives, 34 refrigerant-oil mixtures. 35 The effect of such components as tetraethyl lead, oxidation inhibitors, and aromatic, olefin, and mercaptan compounds on the accuracy of the electrometric determination of water in aviation fuels has been thoroughly investigated. Of the components examined, only mercaptans appeared to show any appreciable interference.36 Ferric ions are reduced by

the Fischer reagent, but the interference may be eliminated by addition to the sample of an excess of 8-quinolinol solution, followed by either direct titration with or backtitration of the Fischer reagent. Satisfactory results were obtained even with such materials as solid ferric chloride and ferric alum.³⁷

A rapid procedure for the determination of oxygen in calcium metal involves the dissolution of the sample in methanol, which does not react with calcium oxide. The water formed by the reaction of the oxide with a subsequently added solution of salicylic acid in pyridine is then dead-stop titrated with Fischer reagent. An interesting variation of the Fischer water reaction has been described by Freedman. He used sulphur dioxide dissolved in pyridine-methanol to titrate N-halogeno-compounds such as chloramine T and N-bromosuccinimide. The dissolved sample is treated with aqueous potassium iodide solution (thereby ensuring the necessary excess of water for this application) and acetic acid, when the liberated iodine is titrated to a dead-stop end point.

Titrations Involving Metallic Ions

Electrode-depolarisation has been used for the determination of silver.39 In two interesting papers, Kies40 has examined the possibilities of mercury electrodes in dead-stop titrimetry. He showed that, with these electrodes, silver, mercurous, and mercuric ions could be used for the determination of halide and certain other ions. Using silver electrodes and an applied e.m.f. of 10 mV., Masten and Stone have described the argentometric titration of chloride, bromide, and iodide, both singly and in succession.41 Iodide has been determined by a divided-cell technique. 42 The solution to be titrated is joined by a potassium nitrate bridge to a saturated solution of mercurous nitrate. A platinum electrode is placed in each solution and connected through a galvanometer. The current, due to the oxidation of iodide, decreases as the iodide solution is titrated with silver nitrate and stops at the end point.

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For the argentometric titration of chloride in dried grasses, Samson used a pair of silver-silver chloride electrodes and an applied e.m.f. of 100 mV.⁴³ The complete titration curve has the form shown in Fig. 6, the current falling to a minimum when the concentrations of silver and of chloride ions are equal. In a later paper, he extended the theory and application of titration with two such second-order electrodes.⁴⁴ Use of silver-silver chloride electrodes in the dead-stop titration of chloride was suggested independently by Bradbury.¹⁶

Keyworth and Stone studied potassium diperiodate cuprate and ditellurate cuprate as analytical exidizing agents, using the dead-stop technique when reaction rates were reasonable. These copper reagents appear to be stable and react almost quantitatively with cyanide and thiosulphate ions. Results were, however, concentration-dependent, and although empirical methods may yield satisfactory results at the milligram level, copper vidations at the millimole level were unsatisfactory for general use.

An indirect method for the determination of mercury involves its precipitation with the highly-selective reagent 2(o-hydroxyphenyl) - benzimidazole. The washed precipitate is dissolved in acetic acid and brominated for about an hour with standard bromate-bromide solution. Potassium iodide is then added and

the liberated iodine is titrated to a dead-stop end point with sodium thiosulphate solution.

Dilute solutions of gallium salts may be titrated with potassium ferrocyanide, preferably at pH 2 and at 50° C.⁴⁷ With an applied e.m.f. of 230 mV., the current remains near zero until an excess of ferrocyanide appears in the solution. Cerate dead-stop titrimetry may be used for the determination of oxalic acid in the microgram range.⁴⁸ In a study of this titration in glacial acetic acid, Hinsvark and Stone showed that although oxidation of one mole of oxalate yields two moles of carbon dioxide, both of the latter come from the solvent.⁴⁹

Vanadium^V in strongly acid solution may be determined by reduction with hydrazine, excess of the latter being back-titrated with standard bromate solution at an applied e.m.f. of 10–20 mV.⁵⁰

Ferric iron may be titrated with ascorbic acid solution. An applied e.m.f. of 140 mV. is used, and the reaction mixture is heated to about 60° C. in the vicinity of the end point.⁵¹ The determination may be made in the presence of such ions as chromium, nickel, nitrate, and phosphate, while small amounts of free nitric acid or of fluoride may be tolerated. Ferrous perchlorate in glacial acetic acid is a satisfactory non-aqueous volumetric reducing agent for the determination of chromium trioxide and of sodium permanganate in the same solvent.⁵² Since oxygen is more soluble in acetic acid than in water, the reagent solution is preferably stored under nitrogen.

Miscellaneous Applications

In the presence of chloroform to extract the liberated iodine, *iodide* in dilute hydrochloric acid may be titrated with standard potassium iodate solution.⁵³ In passing the end point, the current sharply decreases and then increases again. Somewhat high results occur in the presence of bromide; the error may be minimised by allowing about one minute after the addition of each drop of reagent in the vicinity of the end point.

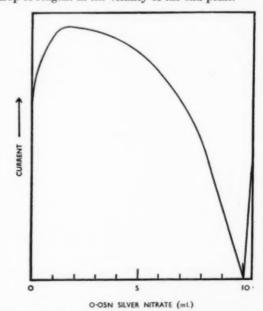


Fig. 6.—Titration curve of chloride obtained with silversilver chloride electrodes.

Monand has described the application of the dead-stop technique to the micro-determination of sulphur in organic compounds by the Zimmermann method.54 The sample is decomposed by heating with potassium, and the resulting potassium sulphide is converted into cadmium sulphide. The determination is completed by the addition of iodide-iodate solution and titration of excess iodine with sodium thiosulphate solution.

Errors in the amperometric and starch indicator methods for the titration of millinormal solutions of iodine and thiosulphate have been investigated. Starch is not only less precise, but also gives blanks which are six to eight times larger than those of the electrical method. 55 The latter has an accuracy of 0.1% or better over the range pH 1-8. In alkaline solutions there

is serious error due to sulphate-formation.

The absorption of iodine by starch⁵⁶ has been studied by an adaption of the dead-stop technique similar to that employed for the iodometric determination of protein sulphydryl groups.⁵⁷ Tanner and Rentschler described the dead-stop iodometric determination of sulphurous acid in intensely-coloured solutions.⁵⁸ In the iodometric dead-stop titration of organic peroxides, the error is less than 0.5% even for a peroxide content of the order of 0.005 milliequivalents.59

Primary amines, such as derivatives of aniline, a- and β-naphthylamines and aminoazo dyes, may be successfully determined by dead-stop titration with sodium nitrite solution in the presence of hydrochloric acid.60 The method is applicable to certain other compounds (e.g., diphenylamine) which react with sodium nitrite in acid solution. According to Stone and Scholten, the end point in this type of application is sluggish compared with other dead-stop titrations.61

A direct bromination to a reversed dead-stop end point may be used for the determination of o-cresol, 4-chloro-2-methylphenol, and 2-methylphenoxyacetic

acid.62

Dead-stop titrations involving acids and bases have been described. 63 According to Mann, quinhydrone is added to the solution, the method being applicable to the determination of acid and saponification values of mineral oils, fats, and waxes in benzene-alcohol solution.64

Small concentrations of oxygen in water (as low as 10-20 y/litre) may be determined by the Winkler method, using thiosulphate titration to a dead-stop end

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United Steel Lectureship in Statistics

THE UNITED STEEL COMPANIES, LIMITED, have agreed to assist the University of Sheffield to create an independent Department of Statistics. Dr. G. H. Jowett, formerly lecturer in statistics at the university, has been appointed head of the new department and took up his new duties at the beginning of the current academic session. Dr. Jowett's post carries the rank of senior lecturer and will be known as "The United Steel Lectureship." Prior to joining the university, Dr. Jowett was a member of the staff of United Steel's Research and Development Department.

Hitherto, the teaching of statistics and research in this field have been the responsibility of the Department of Mathematics at Sheffield University. growth of the subject and the considerable increase in its use in many aspects of scientific and medical work, the establishment of a separate department had become desirable, and was in fact projected as a future development. The new lectureship has made it possible to realise this objective without further delay.

Minister of Education to Open Electrical **Engineers Exhibition**

THE Minister of Education, the RT. Hon. SIR DAVID ECCLES, K.C.V.O., M.P., has kindly consented to open the Sixth Electrical Engineers Exhibition at Earls Court at twelve noon on April 9th, 1957. He will also be guest of honour at the subsequent luncheon, which will be attended by leading figures in the electrical industry. The Minister has shown considerable interest in the special feature stand depicting "Education in the Electrical Industry, Government Services and Educational Institutes.'

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